May 1951 Pages 169-208

The Journal of the SOCIETY OF DYERS AND COLOURISTS

Volume 67 Number 5

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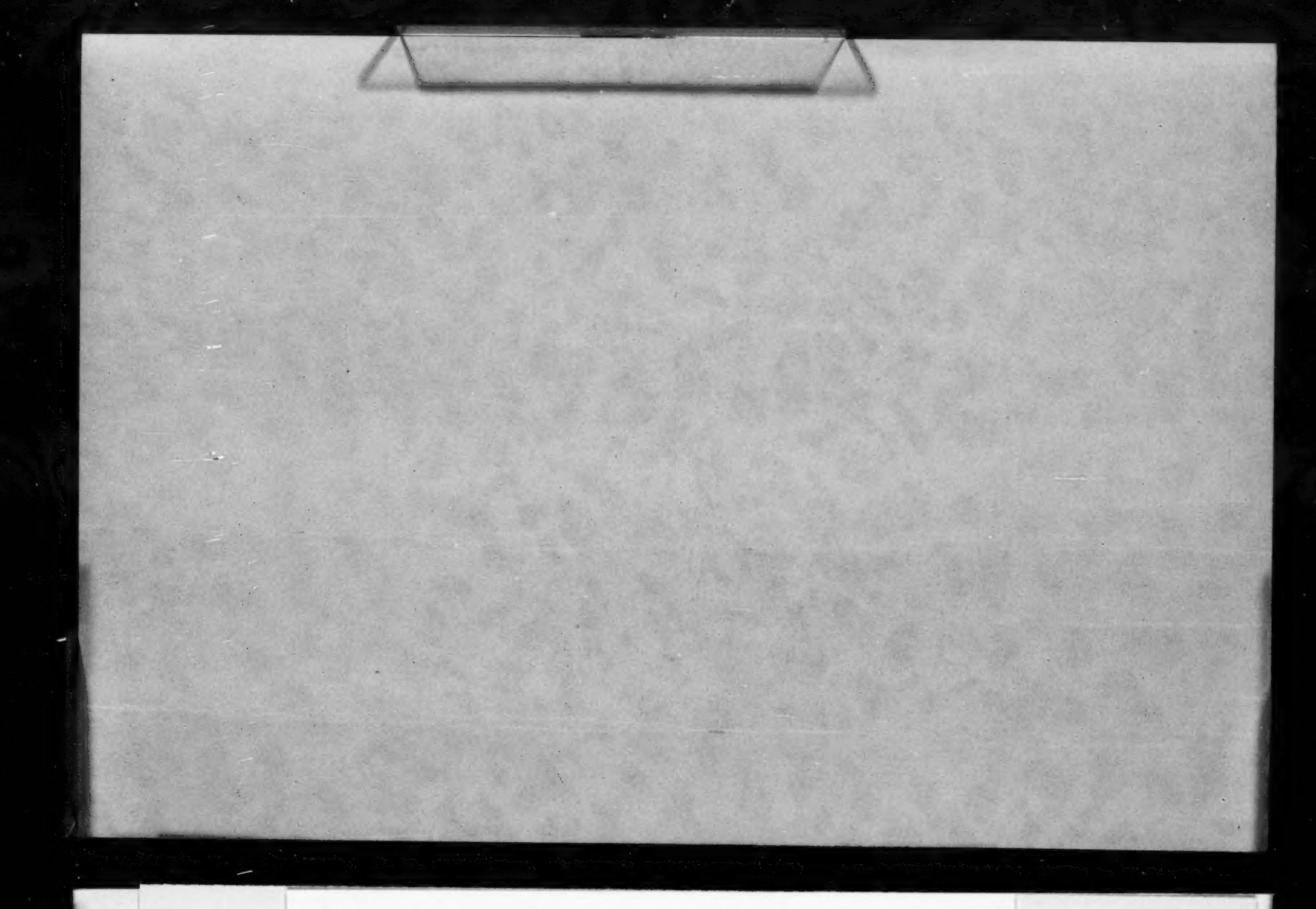
THE SOCIETY OF DYERS AND COLOURISTS 32-34 PICCADILLY BRADFORD YORKS



The Society of Dyers and Colourists

32-34 Piccadilly, Bradford, Yorkshire

Notice and Application Form

INDEX TO THE JOURNAL

Owing to unforeseen difficulties the Index to the 1950 Journal is not likely to be available before July or August.

The Society regrets any inconvenience caused to members and subscribers.

ence on

ARTS TO-DAY

members

22nd September 1951, (Thursday—Saturday), plication of dyes will be reviewed.

re the Conference, and at each of the morning ed by the authors, most of the time being left free n the Saturday. A list of the papers which have

a Reception by the Mayor of Harrogate on the Conference Dinner on the Friday evening.

ated in a region of great beauty and historical wide range of entertainments. Those taking part eir ladies, for whom it is intended to arrange a . The programme of excursions at present being ns, Bolton Abbey, Ilkley, Ripon and Fountains rndale and York.

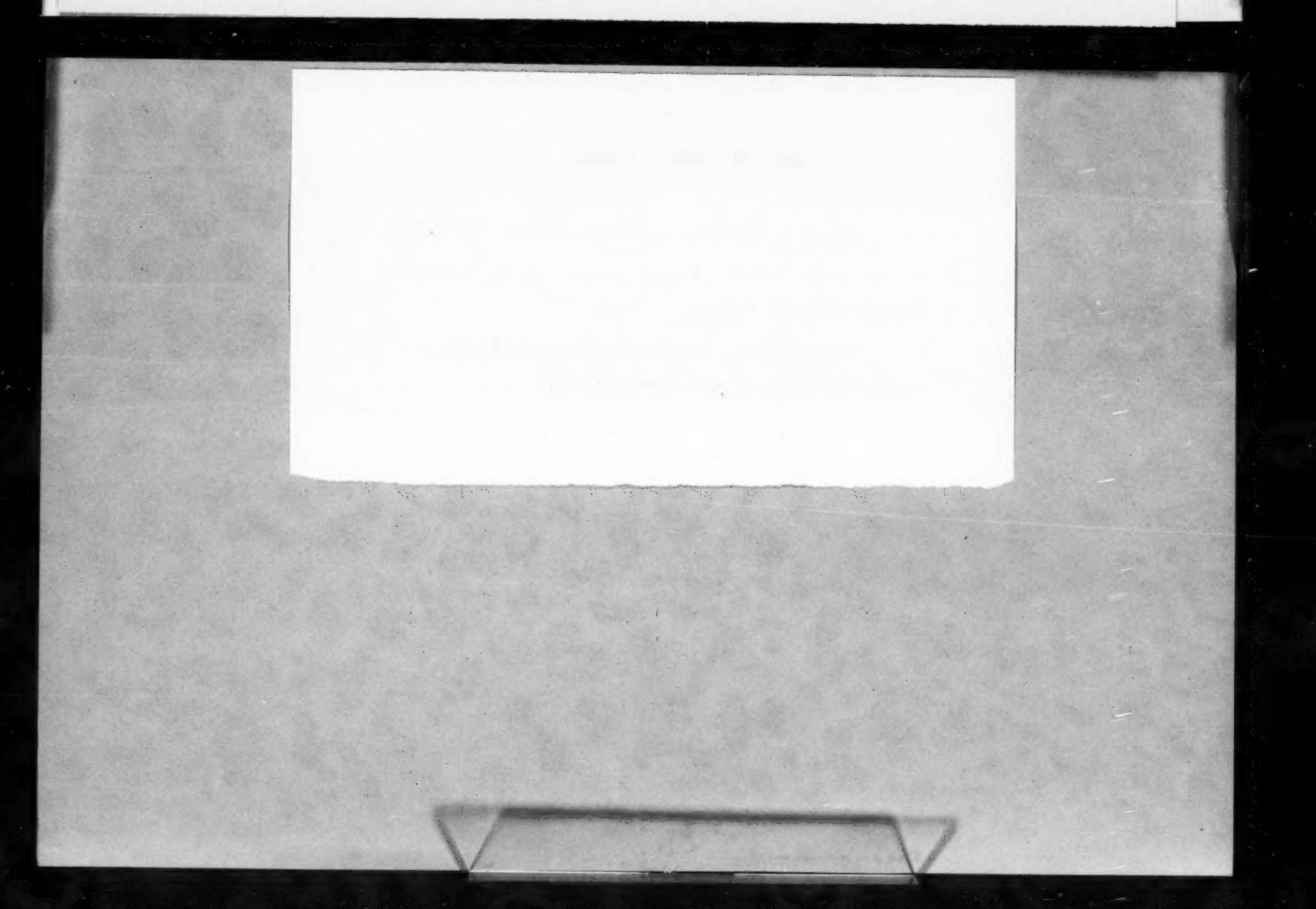
rrogate for the Conference will wish to make their assist them the official list of hotels catering for tion. It is advisable to mention the Conference , however, that hotels may not be willing to allow who experience any difficulty in reserving rooms Secretary, Information Bureau, Harrogate, Yorks.

not payable by ladies accompanying those taking receipt of preprints of papers. A separate charge Bound volumes of papers and discussions will be ber of the Society will be entitled to a single copy

art in the Conference will complete and return the accompanying form without delay.

May 1951

[P.T.O.



The Society of Dyers and Colourists

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A Conference on

THE TINCTORIAL ARTS TO-DAY

Open to Non-members

will be held at Harrogate (Yorkshire) on 20th—22nd September 1951, (Thursday—Saturday), when the evolution of and developments in the application of dyes will be reviewed.

Preprints of the papers will be circulated before the Conference, and at each of the morning and afternoon sessions papers will be briefly introduced by the authors, most of the time being left free for discussion. There will be no afternoon session on the Saturday. A list of the papers which have already been promised is given overleaf.

The provisional social programme consists of a Reception by the Mayor of Harrogate on the Thursday evening (this will include dancing) and the Conference Dinner on the Friday evening.

Harrogate is a well known inland resort situated in a region of great beauty and historical interest, possesses some very fine shops, and offers a wide range of entertainments. Those taking part in the Conference are therefore invited to bring their ladies, for whom it is intended to arrange a special programme of entertainments and excursions. The programme of excursions at present being considered includes:- Harewood House and Gardens, Bolton Abbey, Ilkley, Ripon and Fountains Abbey, Rowntrees of York, Fewston and Washburndale and York.

It is felt that those who intend to stay in Harrogate for the Conference will wish to make their own arrangements for hotel accommodation, and to assist them the official list of hotels catering for conference delegates will be forwarded upon application. It is advisable to mention the Conference when booking accommodation. It should be noted, however, that hotels may not be willing to allow the special conference terms in September. Those who experience any difficulty in reserving rooms are invited to communicate with the Conference Secretary, Information Bureau, Harrogate, Yorks.

The Registration Fee for the Conference (not payable by ladies accompanying those taking part in the Conference) will be £1 and will include receipt of preprints of papers. A separate charge of £1 will be made for the Conference Dinner. Bound volumes of papers and discussions will be available in due course at £1 5s. each, but a member of the Society will be entitled to a single copy at the reduced price of 15s.

It will be helpful if those desiring to take part in the Conference will complete and return the accompanying form without delay.

May 1951

[P.T.O.

Papers to be read at the Conference include the following :

Mr. H. Birchall (Wm. North & Co. Ltd. British Cotton and Wool Dyers Assoc. Ltd.)	*Application of Sulphur Dyes to Cotton Yarns.*
Mr. W. A. Edwards (Wm. Lacey (Loughborough) Ltd.)	*Application of Dyes to Hosiery.*
Mr. J. G. Evans (B.D.A. Ltd.)	*The Prospects before Us.*
Mr. F. Farrington, Dr. P. W. Smith and Mr. F. V. Davis (James Hardcastle & Co. Ltd.)	*Fast Colour Printing.*
Dr. J. F. Gaunt (Patons & Baldwins Ltd.)	*Slubbing Dyeing and Dyestuffs.*
Mr. D. Jagger and Mr. G. Robinson (Carpet Trades Ltd.)	*The Evolution and Development of Dyeing within the Carpet Industry.*
Mr. A. Mellor and Mr. H. C. Olpin (British Celanese Ltd.)	*Developments in the Application of Dyes to Cellulose Acetate Rayon.*
Mr. H. J. Palmer (British Nylon Spinners Ltd.)	*Dyeing of Nylon.*
Dr. W. Shaw (The Calico Printers' Assoc. Ltd.)	*The use of Acetate Colours in Textile Printing.*
Mr. Fred Smith (Wm. Smith & Co. Ltd.)	*Progress of Package Dyeing of Vat Colours and Indigos.*
Mr. W. Thornber (The Silsden Dyeing Co. (1915) Ltd. British Cotton and Wool Dyers Assoc. Ltd.)	*Developments in the Application of Azoic Dyes to Yarns.*
Mr. H. A. Turner and Mr. A. M. El Borai (Manchester College of Technology)	*The Indigosols, Some Observations on their Printing Behaviour.*
Dr. C. M. Whittaker	*Milestones in the Production of Synthetic Dyes for Cellulosic Fibres.*

Papers have also been promised by
Mr. J. Boulton (Courtaulds Ltd.) Mr. J. Baddiley
Mr. D. Howarth (Standfast Dyers & Printers Ltd.)

Assistant Secretary
Society of Dyers and Colourists
32-34 Piccadilly
BRADFORD, Yorks.

Conference on "The Tinctorial Arts To-day"

HARROGATE, 20TH—22ND SEPTEMBER 1951

I wish to attend the Conference, and hereby apply for the items indicated below.

	£	s.	d.	*Number Required*	*Total Cost* £	s.	d.
1. Registration* (including receipt of preprints of papers)	1	0	0				
2. The Conference Dinner on Friday, 21st September 1951	1	0	0				
3. Bound Volume of Conference Papers and Discussions							
(A) Single copy for S.D.C. Members		15	0				
(B) Other copies	1	5	0				
4. Ladies' Programme							
(A) Thursday afternoon, 20th September 1951		10	6				
(B) Friday afternoon 21st September 1951		10	6				

Total remittance enclosed †

5. Invitations to Civic Reception on Thursday, 20th September 1951: Number required

Name (Mr., Mrs., Miss, etc.)
(in Block Letters)

Address
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* Ladies accompanying those attending the Conference need not pay the Registration Fee.

† Cheques should be made payable to the Society of Dyers and Colourists, and should be crossed "Midland Bank Ltd."

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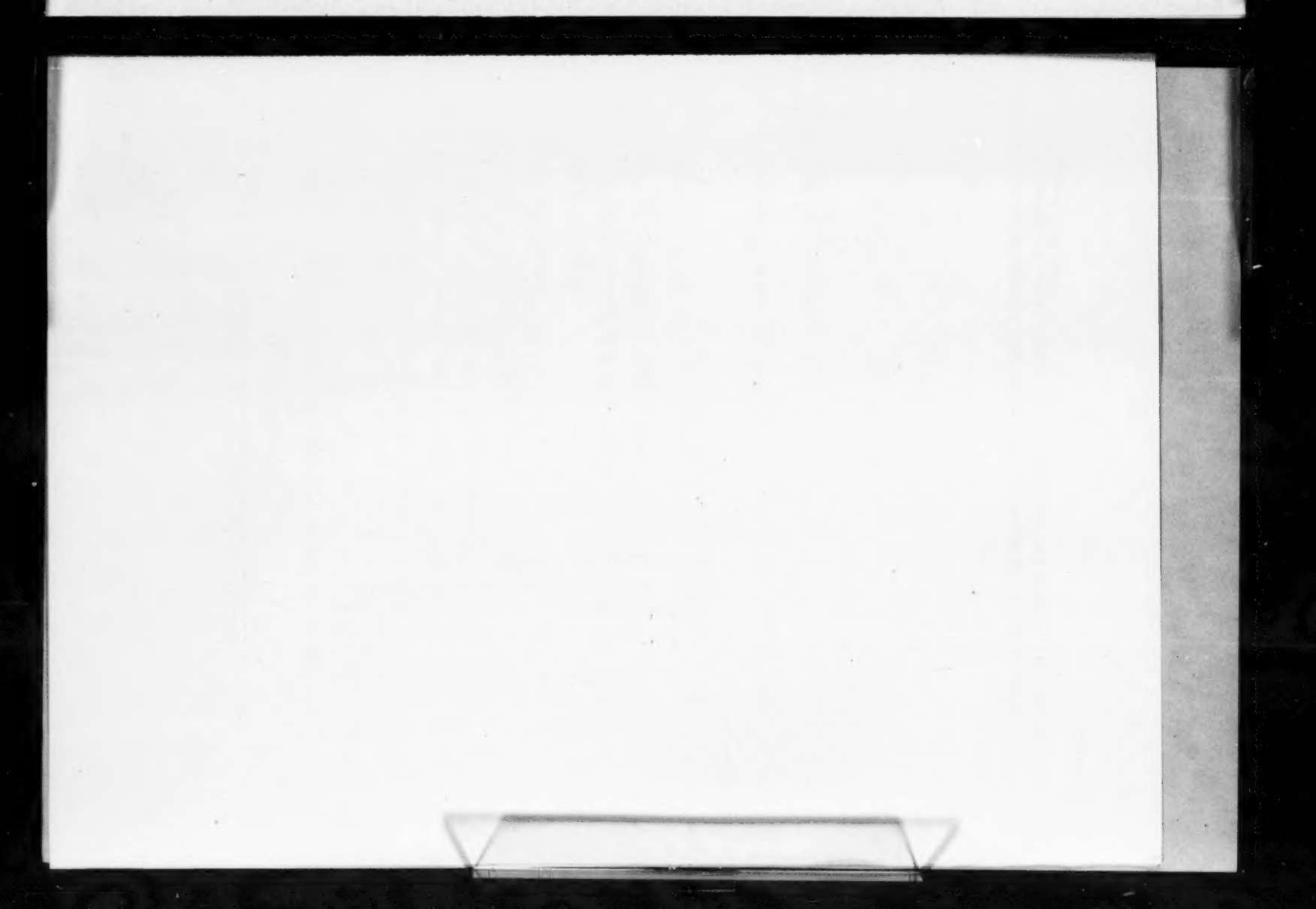

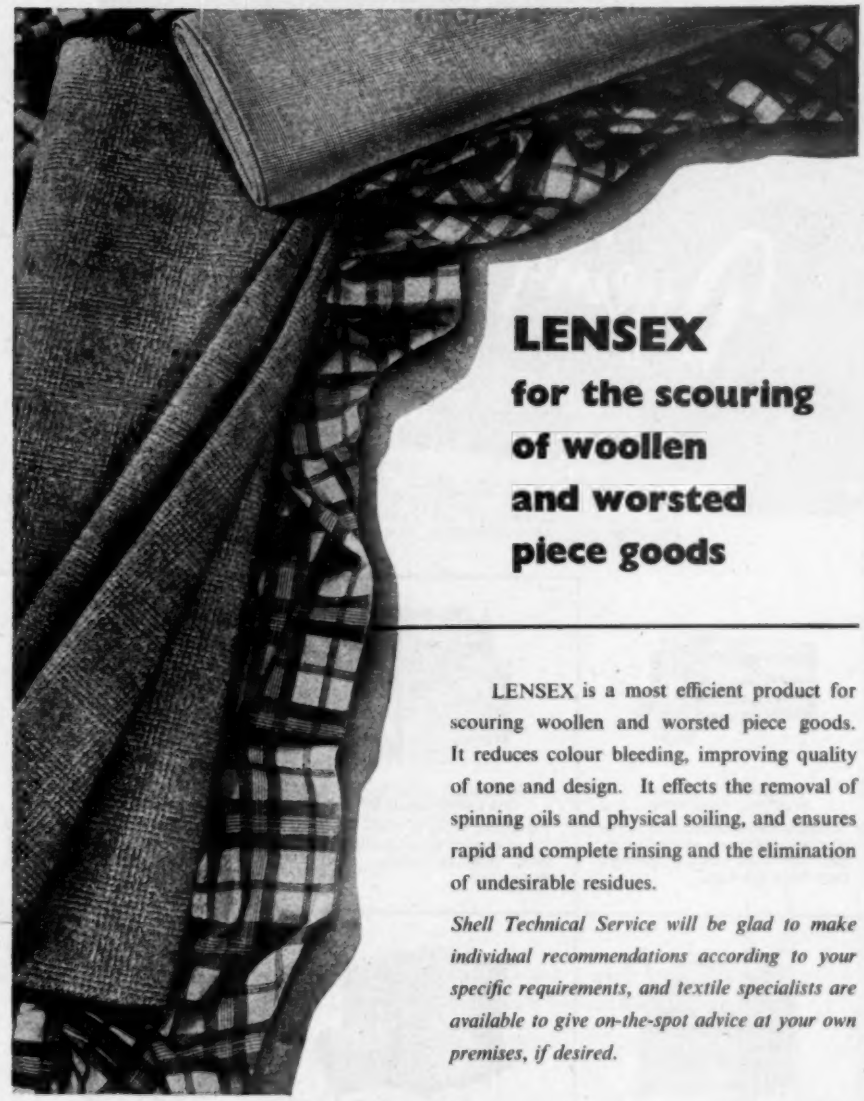

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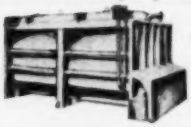

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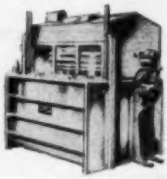

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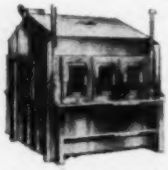

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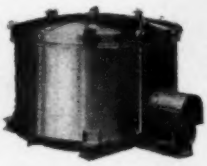

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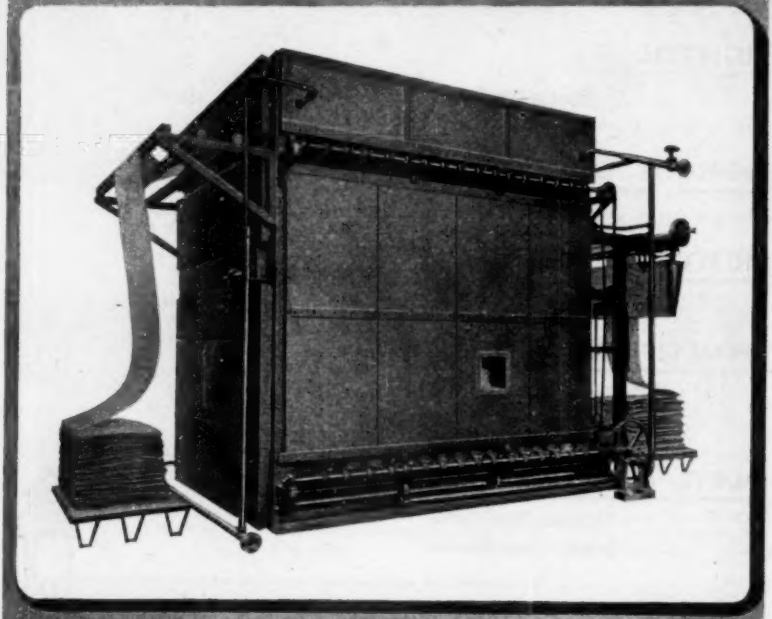

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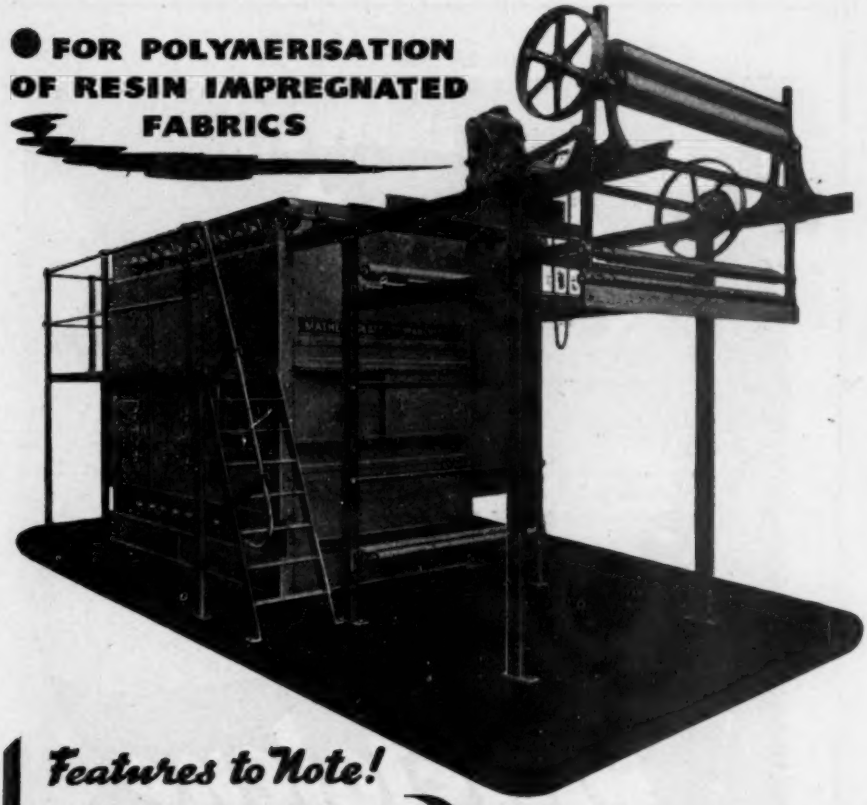

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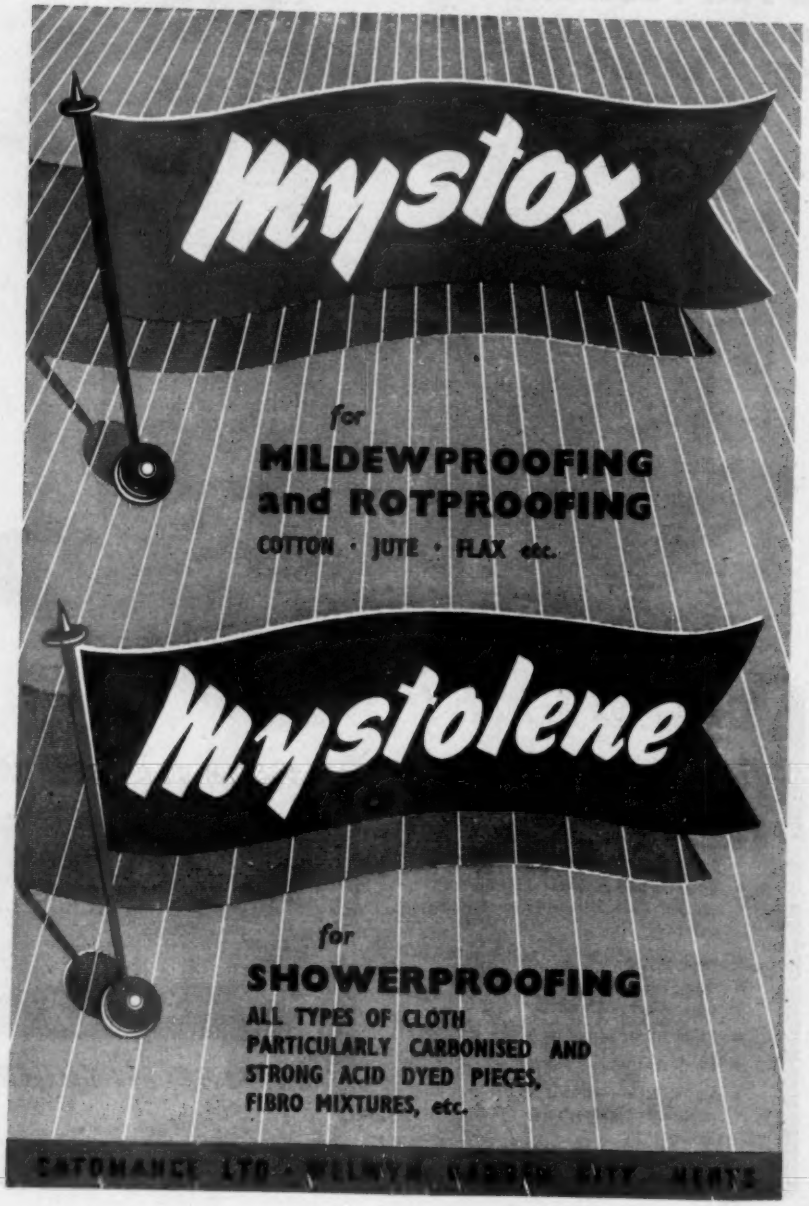
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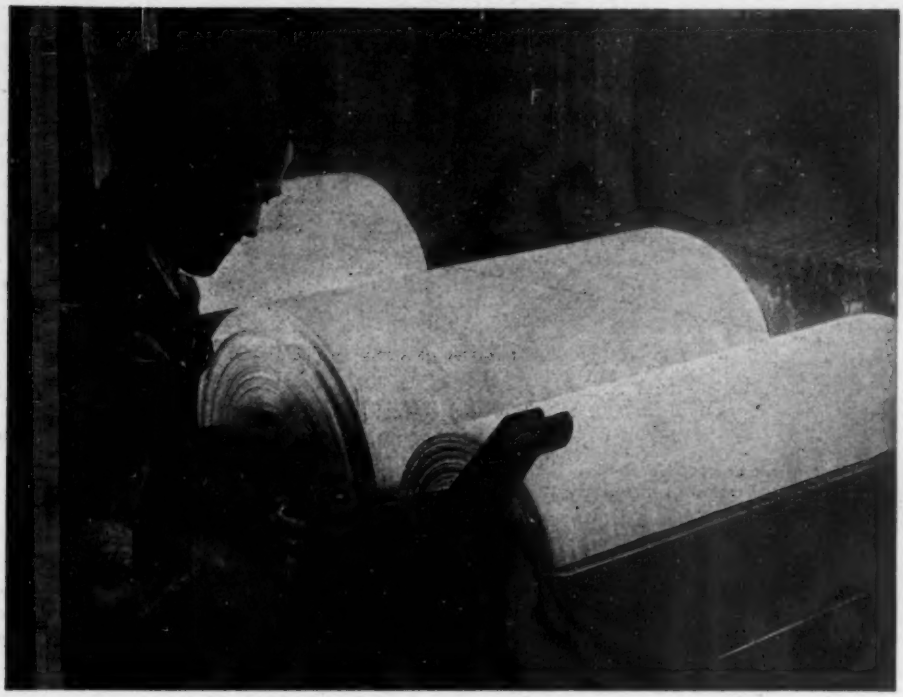

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1951 and pages 345-348 of the July 1950 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 32-34 Piccadilly, Bradford, Yorkshire (*Telephone* Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

MERCER LECTURE

Some Trials by Ingenious Inquisitive Persons— Regenerated Protein Fibres — *D. Traill*

LECTURE

Recent Trends in the Search for New Azo Dyes. IV— Azoic Dyes — *D. A. W. Adams*

COMMUNICATIONS

Reduction of Direct Dyes during the Dyeing of Viscose Rayon — *W. Armfield*

A Study of the Polymerisation of Methacrylamide in Wool — *N. K. Boardman and M. Lipson*

The Preparation and Properties of Regenerated Cellulose containing Vinyl Polymers. I— Internal Deposition of Polymers — *G. Landells and C. S. Whewell*

MEMBERS AND JUNIOR MEMBERS

Persons desirous of joining the Society as Members, Associates, or Junior Members may obtain Application Forms from the General Secretary, or from the Honorary Secretary of any Section of the Society

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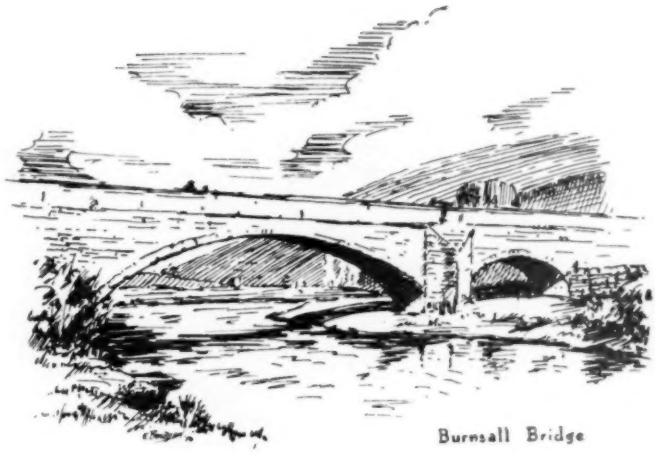

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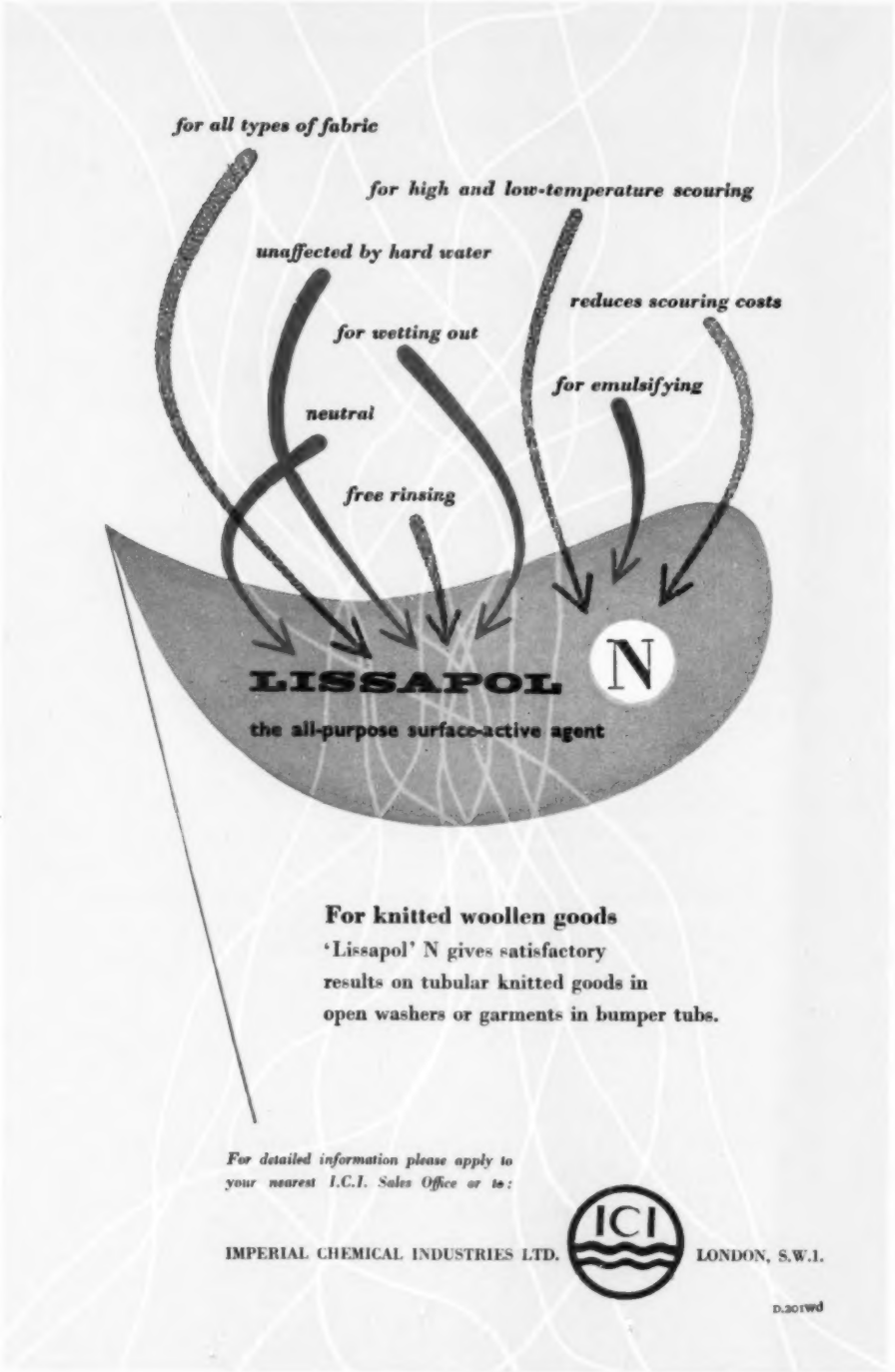

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COMMUNICATIONS

Some Aspects of the Drying and Heating of Textiles
VI—Modifications produced by Thermal Treatments

J. M. PRESTON, M. V. NIMKAR, and S. P. GUNDAVDA

Changes of the properties of fibres after various thermal treatments have been investigated, and it has been found that the amount of change depends on the moisture content and the temperature of treatment. Swelling, moisture absorption, and dye absorption decrease, whilst density increases, as a result of the thermal treatment.

INTRODUCTION

Some information has been published in patents and elsewhere which indicates that thermal treatments reduce the extent to which fibres swell, and that this is true of macromolecular substances in general[1–9]. The published information is far from complete either about the phenomena themselves or about the underlying causes. More knowledge is needed about the behaviour of fibres in particular, because they are regularly exposed to heating, steaming, and drying processes.

It is of interest to know which are the important factors and how far the conditions of treatment affect the result. Furthermore, a change in the fundamental swelling property may be expected to be accompanied by simultaneous changes in other important properties such as dyeing behaviour and wet strength.

In general, the degree of swelling is closely related to the degree of cross-linking[10,11]; consequently, reduced swelling from thermal treatments may be expected to be associated with increased cross-linking. This could result from either chemical or physical causes, e.g. chemical bond formation or increased crystallinity.

Various thermal treatments have been given to two types of regenerated cellulose fibres, viscose and cuprammonium rayon, and to silk, and resulting changes in the fibres have been studied by measuring the swelling, strength, dyeing, density, and other properties.

EXPERIMENTAL

In the thermal treatments given, time, temperature, and humidity were varied. In the first experiments the fibres were freely exposed whilst being heated either in a laboratory oven or in a small steaming cottage. In the later experiments fibres containing known amounts of water were heated in sealed containers, so that the amount of water present remained constant and known. In most of the later experiments samples, each consisting of approx. 3·5 g. of fibres together with a known amount of moisture, were placed in small brass tubular containers each of 45 c.c. capacity which were sealed with rubber gaskets and screw stoppers.

The methods of examination applied to the fibres do not require extensive discussion, since they have been described in previous papers. Measurements of swelling were made using the conductivity method[12], the axial swelling method[13], and the centrifuge method with water[11]. Dry and wet tensile tests were made with a Cambridge Fibre Extensometer. Dyeing of the cellulose fibres was done in a 0·005% solution of pure Chlorazol Sky Blue FF (*C.I.* 518) plus 0·5% sodium chloride at 80°C. in a semi-infinite bath for 2 hr.[14]. The silk fibres were also dyed in Sky Blue FF as described later. Densities were measured in an anhydrous density gradient tube[15]. Some X-ray diagrams were taken with a Philips copper tube.

Moisture absorptions were measured by first drying samples over phosphorus pentoxide, and then conditioning in desiccators over saturated ammonium nitrate solution at 64% R.H. and 18°C. for a week.

DRY HEAT

In preliminary experiments air-dry viscose fibres were heated in a hot air oven for one hour at 100°, 150°, and 170°C. respectively. Subsequently the axial swelling was measured and was found to have undergone a reduction from 7·8 to 7·0 ± 0·1% in each case.

Heating fibres in unsaturated steam may be expected to show similar results, since in this as in hot air the fibres will dry rapidly. Experiments were made in which similar viscose fibres were heated in unsaturated steam at 120°C. for periods

of 10–90 min. The axial swelling was again reduced from 7·8 to 7·0 ± 0·1%, the reduction being effectively independent of the time of heating in this range.

These two sets of experiments suggested that the important stage in the treatment was the initial one before the fibres had dried.

EFFECT OF TIME OF TREATMENT

A new series of experiments was made in which two different viscose fibres were steamed at atmospheric pressure for periods up to 5 hr. The fibres were entered into the steaming cottage in the air-dry condition at room temperature. During the initial heating up of the fibres by the steam there would therefore be a slight condensation, and the initial moisture content would be raised by about 5–10%, i.e. to 20–25% water on the weight of the dry fibres. As will be seen later, this regain approaches the optimum.

The effects of this treatment on the axial swelling and on the water retention after centrifuging are shown in Fig. 1. It is seen that both the axial

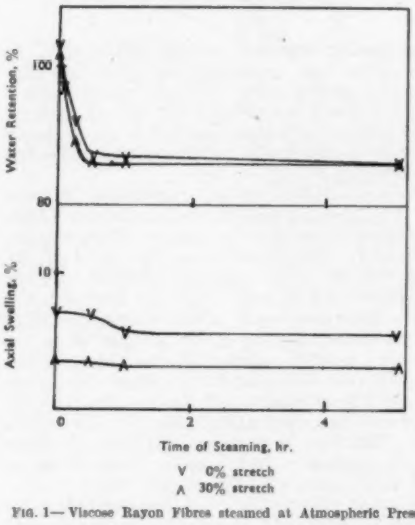


Fig. 1— Viscose Rayon Fibres steamed at Atmospheric Pressure

swelling and the volume swelling as indicated by the water retention [11] diminish more the more prolonged the treatment, and that the separate results obtained with the two different viscose fibres follow similar paths. The 0% stretch viscose, which is less oriented than the 30% stretch viscose, has a much greater axial swelling, and changes in this are, as a result, much more clearly marked, but also the changes are relatively greater.

The water retention and axial swelling curves follow different paths. Initially there is a rapid decrease in water retention, which soon becomes sensibly constant. The axial swelling values show a more gradual decrease, which had not reached a finality by the end of the experiment, though the final changes are too small to show obviously on the graph.

Similar steaming experiments were done with degummed silk with the modifications that fibres with a constant moisture content of 20% were in sealed tubes at 135°C. The changes of water retention and axial swelling with time (Fig. 2) are seen to follow parallel directions to those of the viscose fibres shown in Fig. 1.

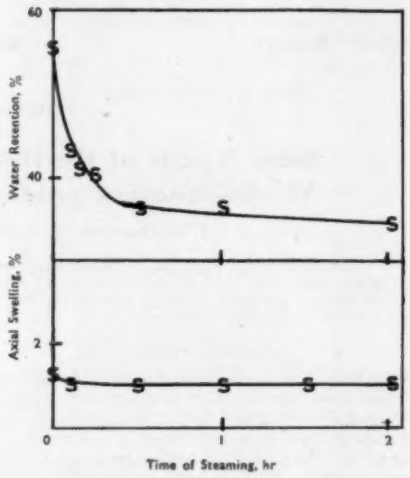


Fig. 2— Silk (20% water) steamed at 135°C.

MOISTURE CONTENT DURING TREATMENT

Statements in the literature had indicated that the moisture content during treatment has a significant effect on the result [2–4]. Series of experiments were made with the 0% stretch viscose fibres and the degummed silk in the sealed brass tubes. The moisture in the fibres in different tests ranged from 0 to 100% or more on the weight of the dry fibres, and the temperature was 120°C. for viscose and 135°C. for silk. The samples supposed to contain 0% moisture had been dried in a vacuum over phosphorus pentoxide for several days.

The water retention and axial swelling values for the viscose fibres are shown in Fig. 3, from which it will be seen that there are well defined minima at 30–40% moisture content.

The densities and equilibrium dye absorptions of these fibres were measured, the results being shown in Fig. 4 and 5 respectively. It will be seen that the position of maximum density in Fig. 4 corresponds to those of minimum swelling in Fig. 3. Again, the minimum of the dyeing curve given in Fig. 5 appears at a corresponding position, where the moisture content during treatment was about 30% on the weight of the dry fibres.

The results of the corresponding experiments made with degummed silk are shown in Fig. 6–8. To obtain the dye absorption curves (Fig. 8), small hanks of silk weighing about 40 mg. were dyed with constant agitation in a semi-infinite bath containing a 0·005% solution of pure Sky Blue FF,

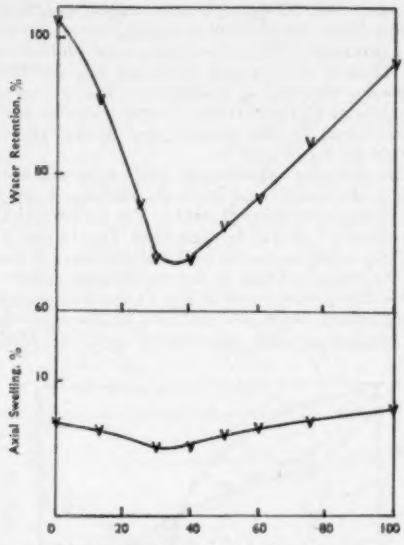


FIG. 3— Viscose Rayon (0% stretch) steamed at 120°c. for 1 hr.

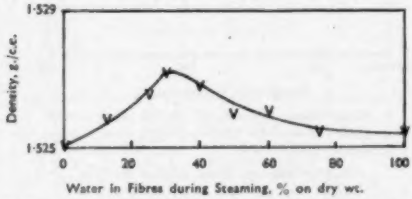


FIG. 4— Viscose Rayon (0% stretch) steamed at 120°c. for 1 hr.

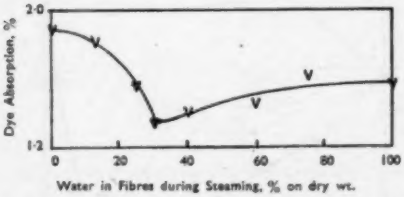


FIG. 5— Viscose Rayon (0% stretch) steamed at 120°c. for 1 hr.

adjusted with acetic acid to pH 3–4, for 2 hr. at 80°c. The amount of dye taken up was determined by stripping the dyed fibres with 25% pyridine as with the dyeings of the cellulose fibres.

These curves show similar trends to those observed with the viscose rayon fibres in Fig. 3–5, but the minima and maxima are not at the same moisture content with the two kinds of fibres. With silk the minimum swelling values shown in Fig. 6 and the minimum dye absorption shown in Fig. 8 as well as the maximum density shown in Fig. 7 all fall at about 20% moisture on the weight of the dry fibres.

TEMPERATURE OF TREATMENT

In addition to the experiments at different moisture contents, other experiments were made in which the temperature was varied between 100° and 175°c. keeping the time of treatment constant at 1 hr. Two different viscose fibres, respectively of 0% and 30% stretch, cuprammonium and silk fibres were used. The experiments with the viscose fibres were done in a steaming cottage at lower temperatures and in an autoclave at higher temperatures. The moisture content during the steaming of the viscose fibres was estimated in the same way as for the experiments of which the results are shown in Fig. 1.

The data for the swelling properties of the viscose fibres are shown in Fig. 9. It will be seen that the water retention falls till it reaches an approximately constant value at about 160°c. The axial swelling values show a different kind of behaviour; they fall up to about 140°c. and subsequently rise steeply.

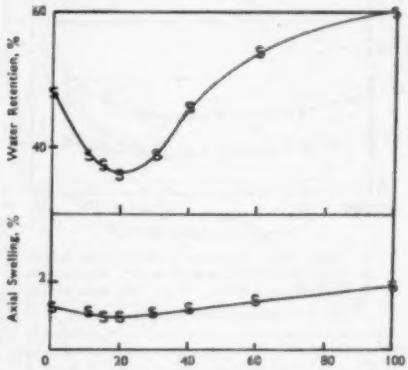


FIG. 6— Silk steamed at 135°c. for 1 hr.

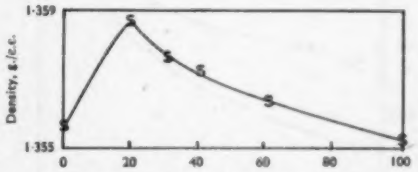


FIG. 7— Silk steamed at 135°c. for 1 hr.

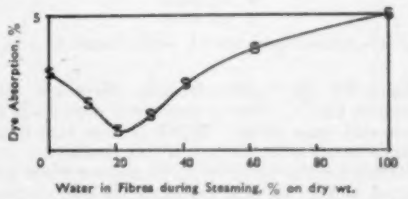
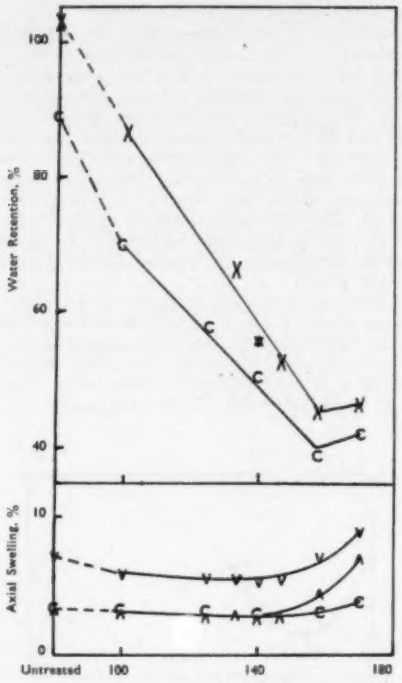


FIG. 8— Silk steamed at 135°c. for 1 hr.



V Viscose rayon (0% stretch), 20–25% water content
A Viscose rayon (30% stretch), 20–25% water content
C Cuprammonium rayon ... 35% water content

FIG. 9— Cellulose Fibres steamed for 1 hr.

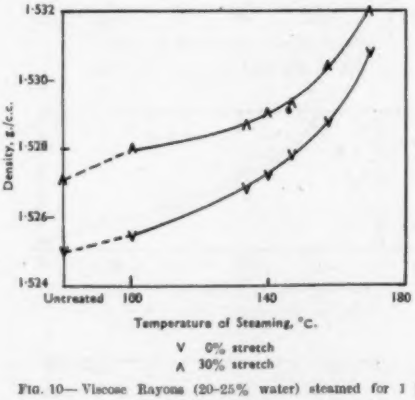
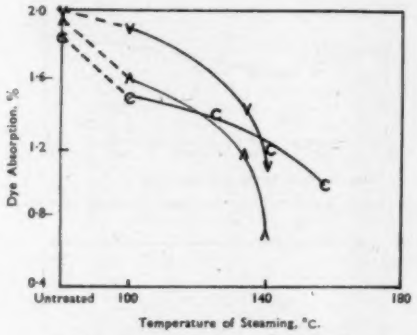


FIG. 10— Viscose Rayons (20–25% water) steamed for 1 hr.

Data for the cuprammonium fibres are also shown in Fig. 9. These experiments were made in the sealed brass tubes. It will be seen that their behaviour is similar to that of viscose fibres.

Values for the densities of the viscose fibres are plotted in Fig. 10, and values of the dye absorptions of the viscose and cuprammonium fibres in Fig. 11. Fig. 10 shows similar trends with both viscose fibres, the density increasing with severity of treatment. The dye absorptions plotted in Fig. 11 show the opposite trend and decrease with increasing severity of treatment. The curves of Fig. 10 and 11 respectively exhibit a similar antibathic trend to the density and dyeing curves plotted in Fig. 4 and 5.

The steaming experiments made with silk were done in the sealed brass tubes, the swelling, density, and dyeing data being plotted in Fig. 12, 13, and 14 respectively. It will be seen from Fig. 12 and 13 that the swelling curves and density curve follow similar paths to those of the regenerated cellulose fibres. The dyeing curve of Fig. 14 up to a temperature of about 140°C. also follows a similar course to the curves for the regenerated cellulose fibres



V Viscose rayon (0% stretch), 20–25% water content
A Viscose rayon (30% stretch), 20–25% water content
C Cuprammonium rayon, 35% water content

FIG. 11— Cellulose Fibres steamed for 1 hr.

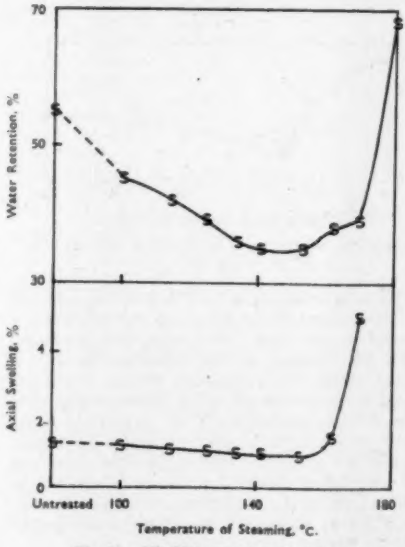


FIG. 12— Silk (20% water) steamed for 1 hr.

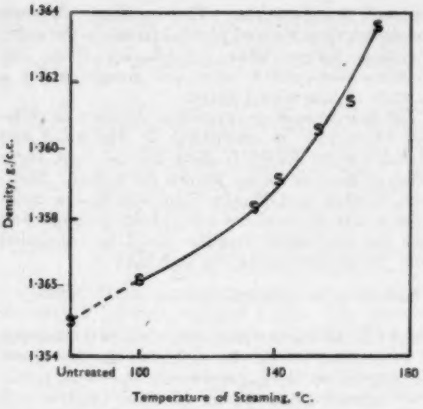


FIG. 13—Silk (20% water) steamed for 1 hr.

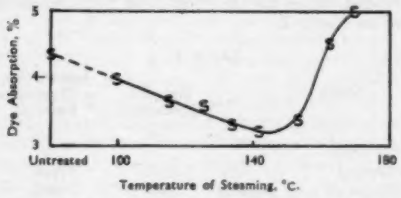


FIG. 14—Silk (20% water) steamed for 1 hr.

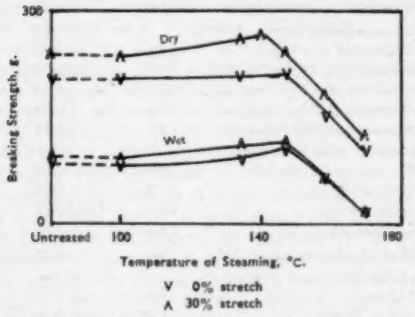


FIG. 15—Viscose Rayons (20–25% water) steamed for 1 hr.

plotted in Fig. 11. The silk data extend to higher temperatures than those for the cellulose fibres, and at the highest temperatures the dye absorption of silk increases to values higher than the initial ones.

Strength tests were made on the cellulose and silk fibres, for the various series in which the temperature of treatment was the variable factor. The data for the wet and dry strengths of the viscose, cuprammonium, and silk fibres respectively are plotted in Fig. 15–17. Fig. 15 shows a slight improvement in both wet and dry strengths with increasing temperature of treatment up to about 130°c. Beyond this point the strengths fall rapidly with further increase of temperature. The strength maxima occur slightly before the minima in the axial swelling curves shown in Fig. 9. The wet strengths of the cuprammonium fibres in Fig. 16 also show a maximum, but this is at a lower temperature than those of the viscose fibres. The dry strengths show no maximum, but the curve takes a distinctly steeper downward trend beyond the maximum of the wet-strength curve. For silk both strength curves of Fig. 17 show only a slow decline at the start but beyond about 140°c. they make a sharp downward bend. This rapid fall of strength is similar to that found with cuprammonium fibres. It occurs at a somewhat lower temperature than that at which there is a sudden change of direction of the axial swelling curve shown in Fig. 12.

X-Ray photographs were taken of untreated viscose fibres and silk for comparison with those

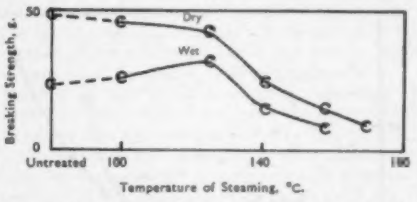


FIG. 16—Cuprammonium Rayon (35% water) steamed for 1 hr.

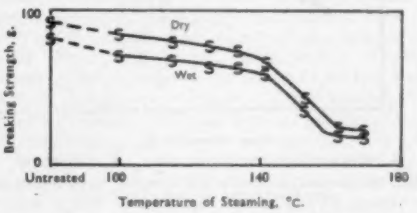


FIG. 17—Silk (20% water) steamed for 1 hr.

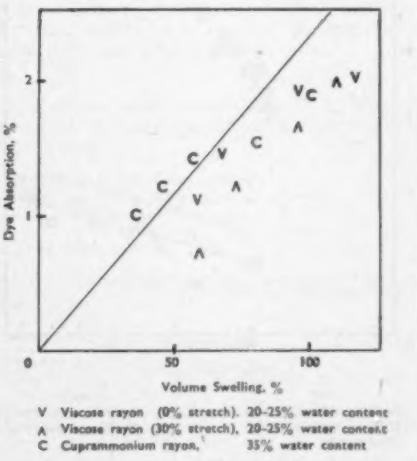


FIG. 18—Cellulose Fibres

that had been steamed at 165°C. On qualitative inspection the X-ray diagrams did not show any significant evidence of change of crystallinity produced by the treatment.

Volume swelling data were measured for some of the cellulose fibres and are plotted against the corresponding dye absorptions in Fig. 18. The swelling values were obtained from the axial swelling data already given and from further measurements by the conductivity method[12].

To study further the changes produced by steaming, the moisture absorptions were measured at 64% R.H. For these measurements sufficient quantities of the silk and cuprammonium fibres remained from the previous steaming experiments, but there was insufficient viscose. New viscose samples were steamed in sealed tubes for 1 hr. with 30% moisture present on the weight of the dry fibres. Different steaming treatments were at different temperatures. The resultant moisture absorption data showed parallel trends to the water retention values after centrifuging (Fig. 19). Moisture absorptions were also measured for a number of unsteamed fibres.

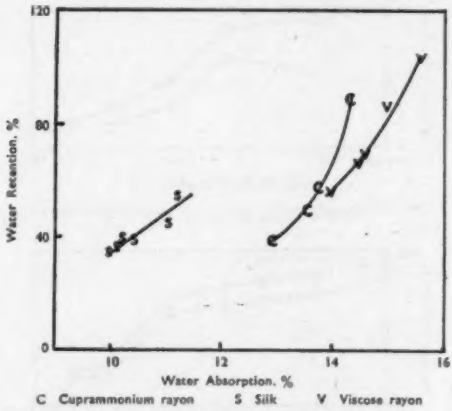


FIG. 19—Water Absorption at R.H. 64% and Water Retention after Centrifuging at 1000 *g* for 5 min.

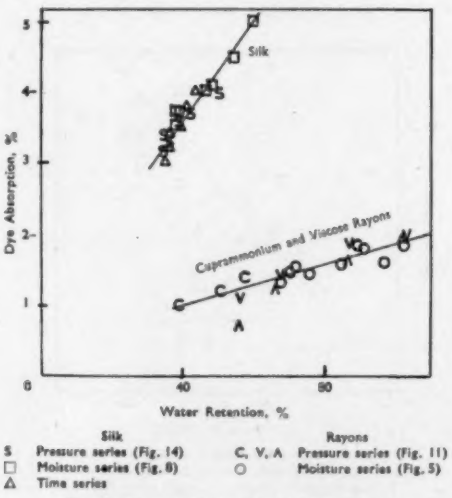


FIG. 20—Dye Absorption and Water Retention

The inaccessible or crystalline fraction of cellulose fibres can be calculated by Hailwood and Horrabin's method[16] if data for at least three different humidities are known for a fibre. However, Preston and Mhatre[17] showed that a mean value could be used for amorphous cellulose and that the crystalline fraction could be calculated from the absorption at one humidity—

$$\text{Inaccessible or crystalline fraction} = \frac{1 - (h/r)_{am}}{1 - (h/r)_{exp}}$$

where h is the relative humidity and r is the amount of water absorbed on the weight of the dry fibres. The expression $(h/r)_{am}$ represents the mean calculated figure for amorphous cellulose, which is 3·09 at 64% R.H.[16]. The measured experimental values were substituted for $(h/r)_{exp}$ in the equation, the calculated values for the crystalline fractions being given in Table I.

TABLE I

Fibre	Moisture Absorption (%)	Inaccessible or Crystalline Fraction
Viscose rayon—		
Untreated	15·5	0·26
Steamed at atm. pressure	14·9	0·29
Steamed at 20 lb./sq. in.	14·5	0·31
Steamed at 30 lb./sq. in.	14·5	0·31
Steamed at 40 lb./sq. in.	13·9	0·34
Cuprammonium rayon—		
Untreated	14·3	0·32
Steamed at 20 lb./sq. in.	13·7	0·34
Steamed at 40 lb./sq. in.	13·5	0·35
Steamed at 70 lb./sq. in.	12·9	0·38
Steamed at 100 lb./sq. in.	12·5	0·40
Untreated viscose rayon—		
No deliberate stretch ...	15·8	0·25
18% stretch	15·5	0·26
30% stretch	15·5	0·26
Tyre cord viscose rayon ...	15·5	0·26
Lilienfeld viscose rayon ...	15·3	0·27
Saponified normal acetate rayon	15·3	0·27
Fortisan (BrC)	11·3	0·46

DISCUSSION

The results obtained in the present work confirmed the statement that there is an optimum moisture content for the maximum modification on heating. Under the conditions used, it was found to be about 30% for the viscose fibres and 20% for the silk fibres examined. Most of the modification was found to occur in the first part of any heat treatment, though the changes continued slowly even after long periods. The series in which the temperature was varied shows that modification increases progressively with temperature, but that there is a useful upper limit set by the marked tendering that occurs at the higher temperatures. This upper limit varied with the different fibres,

but lay in the range 110–130°c. under the conditions used. It is, of course, possible that it would have lain at higher temperatures for shorter periods of heating.

In the extreme cases thermal treatments had great effects on the swelling properties of the fibres, which were reduced by about a half with the cellulose fibres and by about a third with silk. There were corresponding changes of density amounting to an increase of about 0·005 for the viscose fibres and about 0·008 for silk. The greatest density observed for viscose, 1·532, although significantly higher than the value for the corresponding untreated fibres, is still a long way below the densities of native cellulose fibres. The greatest increase of density found with cellulose fibres corresponds to an increase in crystallinity of about 4% according to the relation between density and crystallinity discussed by Preston and Mhatre[17]. This small increase of crystallinity appears totally inadequate to account for the great reduction in swelling when considered in relation to the water retention values and crystallinity of cotton or linen[11,17]. This view is not contradicted by the X-ray evidence. A similar conclusion may be true of silk, but comparative density and crystallinity data are lacking for a range of silk fibres. The conclusion is probable, as the density changes are only slightly larger than those of the regenerated cellulose fibres, and the small amount of X-ray evidence again does not contradict it.

The data for the moisture absorptions given in Table I indicate that steaming produces higher increases of "crystallinity" than are indicated by the corresponding increases of density. The increases of crystallinity, even taking the higher values derived from the moisture absorption measurements, are still inadequate to account for the greater reduction in swelling produced at the same time when considered in relation to the water retentions, moisture absorptions, and crystallinities of native cellulose fibres.

If major crystallinity changes have to be excluded as the main factor in explaining the profound modifications introduced by thermal treatments, there still remains the possibility that the cause lies in cross-linking due to chemical bond formation, e.g. to ether links[18] or physical bonds caused by small local crystallisations. The observation that the optimum condition is one of appreciable moisture content suggests that the latter is the more probable explanation. The comparatively minor improvement in wet strength shown by the regenerated cellulose fibres also agrees with a minor change in crystallinity when compared with the strength changes that occur on wetting the native cellulose fibres.

The data for all the fibres show that the progressive reduction in swelling, as shown by the changes of water retention, is at first produced by a simultaneous, and for the viscose fibres very approximately equal, reduction of both axial and transverse swelling values; but with the most severe treatments the axial swelling actually increases whilst the water retention continues to decrease, and correspondingly there is a marked loss of strength. These observations suggest that there is first a general increase in bond formation roughly in proportion to the distribution of fibre molecules, but in the last stages increased cross-linking is accompanied by decreased axial bonding resulting from depolymerisation of the cellulose. In the initial stages the effect of the former preponderates, whilst in the later stages it is the latter which is the more important.

The data of dye absorption like those of water retention show profound changes. The regenerated cellulose fibres show reductions of as much as a half and the silk fibres of as much as a third. It was shown previously[19] that there was a relation between the volume swelling of viscose fibres, spun under different conditions, and the equilibrium dye absorptions. The data obtained in the present work have been examined from this point of view, and dye absorptions plotted against water retention values in Fig. 20. Each series of data shows a similar relation: there is seen to be a decrease of dye absorption with decrease of weight swelling as indicated by water retention*.

In Fig. 18 the continuous curve represents the theoretical relation between dye absorption and the volume of the internal phase[19,20]. The points lie about the curve, and each kind of fibre shows a progressive decrease of dye absorption with decrease of swelling. It is interesting to note in both Fig. 18 and 20 that under these conditions of dyeing there are no significant differences of dye absorption between cuprammonium and viscose fibres.

In all the experiments in which the samples of moist fibres were heated there were inevitably moisture transfers due to temperature gradients set up[21,22]. As moisture content is an important factor which affects the swelling, dyeing, and density of the resultant fibres, it is to be expected that any ordinary heating treatments will lead to some local irregularities. These will be greater the thicker the masses of fibres involved. In the present work the attempt was made to minimise these irregularities by using small samples in thin layers, but it would be too optimistic to hope that they have been eliminated entirely, and some of the irregularities in the data may arise from this cause.

* * *

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* Data for the very highly degraded silks steamed at 162°c. and 170°c., which had disproportionately high dye absorptions, were not plotted.

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The Combination of Wool with Acid Dyes
I—An Automatic Photometer for the Measurement of Rates of Dyeing

N. H. CHAMBERLAIN and G. H. LISTER

An automatic photometer for measuring the rate of absorption of dyes from solutions of constant concentration is described, and its use illustrated. It is shown that photometric control of dye concentration in solutions of free dye acids and at pH below approx. 3·5 is not feasible, owing to hydrolysis of the wool sample by the dye liquor; alternative means of control by electrical conductivity are described. When the correct means of control are applied, the sensitivity and accuracy of the apparatus, and hence the reproducibility of the results obtained, are of a high order.

A new type of D.C. amplifier circuit is incorporated in the photometer, which can be almost perfectly stabilised towards variations in the mains supply voltage, without the use of constant-voltage transformers or other external voltage-regulating devices.

1. INTRODUCTION

In order to facilitate experimental work on the mechanism of dye absorption by textile fibres, and to simplify the interpretation of the results obtained in such investigations, there has been apparent for some time the need for an experimental technique which would enable the rate of uptake of dye by such materials to be measured in solutions of constant dye concentration. One suggested method for achieving this result consists in dyeing the samples in a very large excess of the dyebath, so that the amount of dye removed by the samples is negligible compared with the total amount present. The absorbed dye is then stripped from the dyed samples by means of aqueous pyridine, and estimated either photometrically or by titration with titanous chloride. It is obvious that if the dye absorbed is to be determined as a function of the time of dyeing, one sample will have to be dyed for every point required on the rate curve, and while these may all be dyed together in the same bath, and removed at appropriate intervals (providing the liquor ratio is maintained at a value adequate to justify the basic assumption that only a negligible fraction of the total dye is removed from the bath by all the samples entered), each must be dealt with individually for extraction and estimation of the dye absorbed. If many points are required on the rate curve, and many such curves have to be plotted, the labour involved becomes fairly formidable.

In the instrument to be described here the alternative principle is used, namely that of dyeing a single sample in a dyebath at a liquor ratio of about 100 : 1, and maintaining the concentration of the dyebath constant by adding to it, as dyeing proceeds, additional concentrated dye solution from a burette, the addition being automatically controlled by a suitable photoelectric relay circuit so that no appreciable departure from the original concentration is at any time permitted. It is then plain that a set of burette readings taken at various times will enable the rate curve to be directly plotted. The method has several advantages: large quantities of purified dye are not consumed in making up excessively large dyebaths, and large amounts of the material to be dyed are also unnecessary, since the whole rate curve is obtained from one sample. These considerations are by no means negligible, since the purification of dyes and substrates often occupies a considerable proportion of the total effort expended in such work. Possible experimental error in the extraction and estimation of the dye absorbed by the samples is also avoided.

2. GENERAL PRINCIPLES AND DESIGN OF THE INSTRUMENT

A sample (approx. 2 g.) of the wool is dyed in a circulating dyebath at a liquor ratio of approx. 100 : 1. The vessel containing the sample and dye liquor is designed so as to enable a beam of light to be passed through a definite thickness of the solution while dyeing is in progress; this beam of light is received by the cathode of a photocell, termed the *test* cell. At the same time, a similar beam of light, derived from the same source, is passed through an equal thickness of the original dyebath, contained in a small glass cell, and falls on a second photocell, the *standard* cell. The outputs from both cells, after amplification, are combined in series opposition, and conditions are so adjusted that, when the light transmission of the two solutions is the same, the net output is zero. As the sample absorbs dye, the light transmission of the test photocell no longer exactly balances that of the standard cell, and the combination has a definite voltage output which increases with increasing absorption of dye. This voltage is applied to the grid of a gas-filled relay, and causes the latter to conduct at some predetermined (small) departure from zero output. When the thyratron conducts, a secondary relay in its anode circuit opens the electrically operated valve of an automatic burette, which delivers concentrated dye solution into the vessel containing the sample, thus restoring

the circulating dyebath to its original concentration. When this state is reached, the net output of the twin amplifiers again becomes zero and the thyratron ceases to conduct, thus stopping the flow from the burette. The amount of dye absorbed by the sample after any given time is obtained directly from the burette reading. If the apparatus is sufficiently sensitive, the concentration of the test solution can never depart significantly from the original value, so that dyeing is carried out from a solution of constant dye concentration. Constructional details of the apparatus are given below, the description being illustrated by Fig. 1–8.

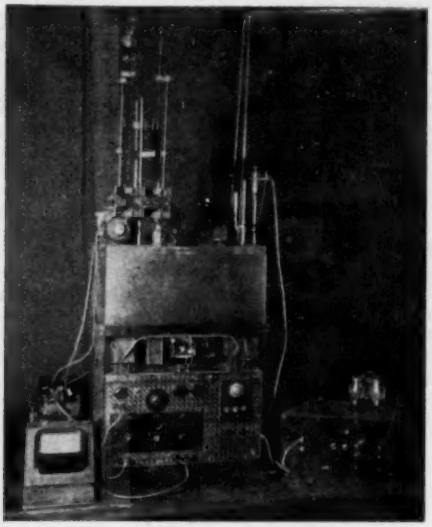


FIG. 1

Fig. 1 shows a general view of the complete apparatus as finally constructed. The upper section of the main assembly consists of a thermostat bath in which the tube containing the sample and dye liquor is clamped. The space underneath the tank is occupied by the amplifier and relay unit, and the light source and photocells are placed above the amplifier unit and immediately below the tank. (The opening above the amplifier unit is normally closed by means of a close-fitting sheet metal cover to exclude stray light; this has been removed in the photograph to enable some details of the internal arrangements to be seen.) To the right of the main assembly is a small thyratron unit used for controlling the thermostat. It combines the functions of a sensitive speed control for the stirrer motor, which is located behind the main assembly, and a relay operated by the thermoregulator to switch on and off an immersion heater in the tank. The advantage gained by using a thyratron as the primary relay for this purpose is that the power required to operate it is so minute that there is no sparking whatever at the regulator contact, with the result that the mercury surface in the latter remains clean indefinitely; this is of importance in the present case, since the on–off cycle of the regulator is only about 1 min. when the thermostat is controlled at 60°C., and such heavy duty would soon render it ineffective were any sparking to occur. The regulator itself is of conventional type, but is entirely filled with mercury, so that it can be used up to temperatures of 100°C. if necessary. Since, however, the thermostat control gear is only ancillary to the main apparatus, and control of the tank temperature could be equally well obtained by any of the standard methods, it need not be further described.

On the left of the main unit in Fig. 1 is seen the output voltmeter used for initial balancing and standardisation, and behind it the 2-v. accumulator supplying power for the electrically operated valves of the automatic burettes. These latter, together with the driving gear for the circulating pump of the sample tube, are mounted on a platform covering part of the left-hand end of the tank; and the thermoregulator is seen in position, together with a thermometer, in the right-hand front corner. The two vertical tubes rising behind the thermoregulator conduct water to and from the water-jacket surrounding the light-source, the inlet and outlet of the latter being situated at the back of the main unit.

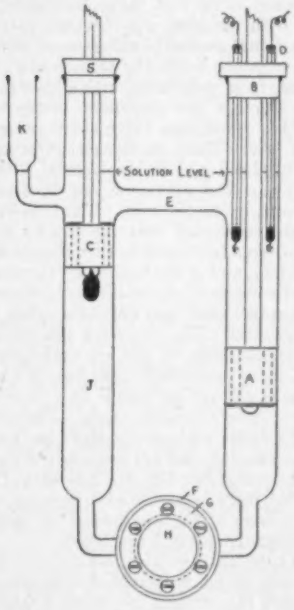


FIG. 2

The individual parts of the apparatus may now be considered in greater detail. The sample to be dyed is placed in the left-hand leg *J* of the U-tube shown in Fig. 2. Above it, in the same leg, is a cylindrical polythene plug *C*, supported by a

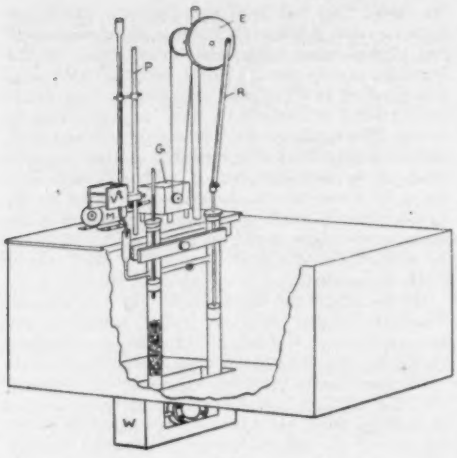


FIG. 3

thermometer which is itself held in position by the rubber stopper *S*. The plug is pierced vertically by six equidistant holes, which are closed at the upper surface of the plug by a thin flexible diaphragm cut from polythene foil. This diaphragm is held down at the centre only, and thus constitutes, in conjunction with the plug, a non-return valve, which has been found perfectly effective in practice.

In the opposite leg of the U-tube is a piston *A*, again made from polythene, with a glass piston-rod working through the polythene stopper *B*, and fitted with a non-return valve of the same type on its lower face. Thus, as the piston is moved up and down, the liquid in the U-tube is circulated positively in a clockwise sense, and does not merely surge to and fro in the tube. The two legs of the U-tube are connected near the top by the cross-tube *E*, of large bore so as not to impede the circulation unduly, and at the bottom by the test-cell *H*. The latter consists of an annular polythene housing *F*, with parallel sides into which two glass windows *H* are recessed. The windows are retained by brass covering-rings *G*, one on each side of the housing, and a water-tight seal between glass and polythene is ensured by means of a narrow gasket of thin polyvinyl chloride sheet. The glass inlet and outlet tubes are made a tight push-fit in the polythene housing, and are assembled with a smear of Bostik cement No. 771 (BB Chemical Co. Ltd.). No trouble from leaks has ever been experienced. The inner faces of the windows *H* are arranged to be very approximately 1 cm. apart.

The U-tube also possesses a side-tube *K* for the introduction of concentrated dye solution during dyeing, and the stopper *B* carries a pair of electrodes *D*, used for conductivity control as described later; these consist of short lengths of 22-s.w.g. platinum wire fused into soda-glass tubes, connection with the platinum inside the tube being made with mercury in the usual manner. The capacity of the whole U-tube, up to approximately the level indicated in Fig. 2, is 200 c.c.

Fig. 3 shows the U-tube, together with its accessories, in position in the thermostat. The latter possesses a rectangular well *W* into which the test cell drops, and which is also fitted with glass windows, so that a beam of light may be passed through the test-cell while it is in position in the tank. The piston is driven from the eccentric *E* by means of the connecting-rod *R*, power being obtained from the small motor *M* (0·013 h.p.) through the worm reduction unit *G*. The piston normally operates at 40 strokes per minute, and since approx. 25 c.c. of liquid is displaced at each stroke, the whole of the liquid in the U-tube is forced through the sample approx. 5 times per minute.

A somewhat similar arrangement has been used by Davenport[1] as the basis of a photometer designed to measure the rate of exhaustion of a dyebath during the dyeing of a small sample of fibre. His apparatus differs from the present one in that no attempt was made to control the concentration of the dyebath during dyeing, but the amplifier used was given a logarithmic characteristic, so that the readings of the output meter were linearly related to dye concentration. Also, Davenport used a revolving impeller to circulate the liquor through the sample. In view of his own finding that the rate of dyeing is critically dependent on the rate of circulation, this seems unwise, since such impellers are not positive in action as a piston is. The type of material dyed is not stated; in the present case, for wool samples, it was found, as will be shown later, that the rate of absorption of dye is almost independent of rate of circulation over a wide range.

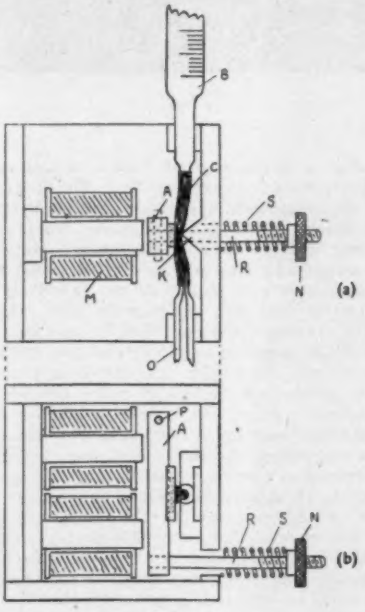


FIG. 4

The automatic burettes (of which only one is shown in Fig. 3 though two are actually fitted) are clamped to a vertical rod *P* in a position convenient to allow them to discharge into the side-arm of the U-tube (*K*, Fig. 2) when the electrically controlled valve *V* is operated by the amplifier and relay system.

Details of the valve *V* are shown in Fig. 4, in which (A) is a vertical section and (B) a plan of the device. The burette *B* and the outlet tube *O* are rigidly clamped in line with one another, and connected by a short rubber sleeve *C* consisting of cycle valve tubing. This rubber sleeve is normally compressed between a blunt ebonite knife-edge *K* and a small ebonite block attached to the face of the armature *A*, which pivots on a pin *P* and is spring-loaded against the knife-edge by means of a rod *R*, passing through a clear hole in the back of the housing and carrying a compression spring *S* adjustable as to tension by the milled nut *N*. In this state, therefore, flow from the burette is cut off. When the electromagnets *M* are energised, however, the armature *A* is pulled off, and allows the rubber sleeve to open, so that the burette delivers liquid at a rate determined mainly by the bore of the capillary outlet *O*. On breaking the magnet circuit, the flow stops immediately. The magnets are wound with 30-s.w.g. double-silk-covered wire to a resistance of 13·3 ohms each, and the two are operated in parallel from a 2-v. accumulator. The total resistance is thus 6·6 ohms and the operating current approx. 300 ma.

Operation from a D.C. source is preferred because the magnet cores are not laminated, and would heat up if run from an A.C. source.

The optical system of the apparatus is shown in Fig. 5. The light-source *l*, with its coil filament *f*, is situated symmetrically beneath the tank, surrounded by a cylindrical water-jacket *wj* and enclosed in a metal housing *lh*. The lamp used is an 8-v. 4-amp. projector lamp (Osram type EL 1) and is run at only 6·3 v. Longer life and decreased sensitivity to voltage variations were thereby obtained, while the light output, though decreased, remained adequate. The sides of the lamphouse have rectangular apertures cut in them, and are fitted with metal slides, having long wedge-shaped openings longitudinally placed, as shown in Fig. 5(B). By moving these slides, the intensity of light in the two beams can be varied at will. The test cell *tc* is shown in section in its position in the well *w* of the tank, and the standard cell *sc*, containing the original dye solution, is supported as shown in a corresponding position in the other beam. In practice this cell is fitted with an air-tight cover to prevent evaporation and consequent increase of dye concentration during long runs. The two photocells *pc* are also enclosed in metal housings *ph*, having apertures in the sides facing the light source. Colour filters may be inserted as required to cover these apertures. For fine adjustment purposes, each photocell housing has mounted within it a small flash-lamp bulb (4·5 v.), which is connected, in series with a 50-ohm variable resistance, to the 4-v. filament supply of the amplifier valves. This enables the lamps to be turned down to a barely visible glow, and provides for sensitive adjustments of the amount of light reaching the photocell cathodes. No lenses are used in the optical system, the two light beams being completely divergent, and limited in area by the slides of the lamphouse. The beam on the test side passes through a certain thickness of thermostat water in addition to the test solution, and it might be supposed that errors would be introduced by suspended dirt in the tank. In actual practice the tank is filled with distilled water to start with, and remains very clean, possibly because the temperatures at which it is normally run (50–80°C.) are too high for bacterial fouling to occur. No trouble has in fact been encountered from this source.

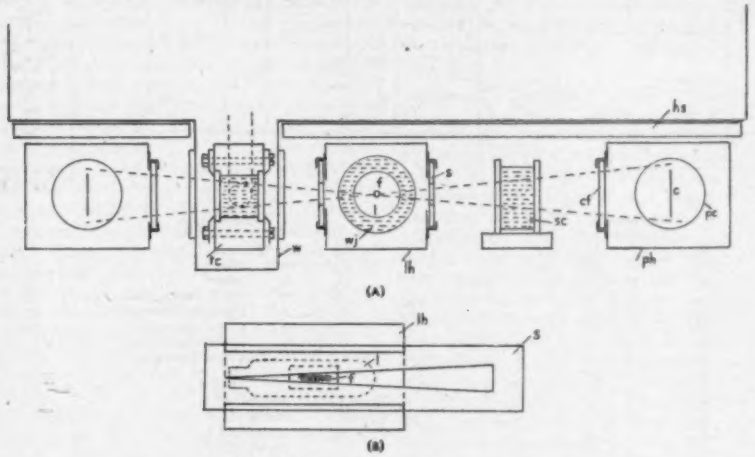
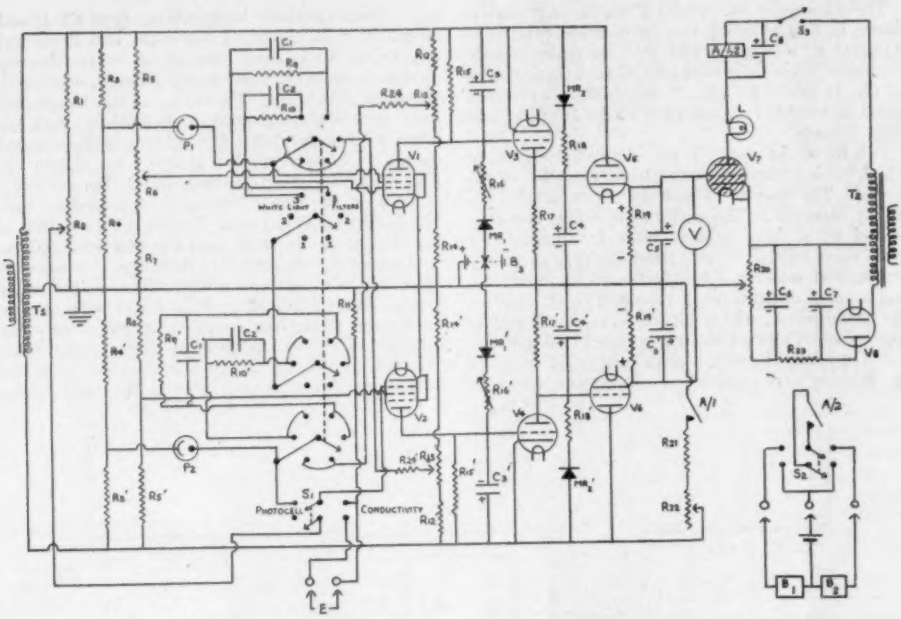


FIG. 5



Resistances (ohms)		
R_1	...	34×10^3 (2 w.)
R_2	...	10×10^3
R_3	...	120×10^3
R_4	...	50×10^3
R_5	...	50×10^3
R_6	...	10×10^3
R_7	...	30×10^3
R_8	...	35×10^3
R_9	...	150×10^3
R_{10}	...	30×10^3
R_{11}	...	1×10^3
R_{12}	...	270×10^3
R_{13}	...	1×10^3
R_{14}	...	1×10^3
R_{15}	...	250×10^3
R_{16}	...	100×10^3
R_{17}	...	68×10^3
R_{18}	...	47×10^3
R_{19}	...	47×10^3
R_{20}	...	5×10^3
R_{21}	...	6×10^3
R_{22}	...	5×10^3 (5 w.)
R_{23}	...	5×10^3
R_{24}	...	120×10^3

Capacitances (microfarads)		
C_1	...	0·003 (mica)
C_2	...	0·01 (mica)
C_3	...	2 (paper)
C_4	...	8 (350 v. electrolytic)
C_5	...	8 (350 v. electrolytic)
C_6	...	25 (50 v. electrolytic)
C_7	...	50 (50 v. electrolytic)
C_8	...	50 (50 v. electrolytic)

Valves		
$V_1 = V_2$	...	Mullard EF 6
$V_3 = V_4$	...	Osram ML 4
$V_5 = V_6$	...	Osram MH 4
V_7	...	Osram GT1C
V_8	...	Osram D 42

$A/1, 2$	...	250 ohms resistance
B_1	...	6·6 ohms resistance
B_2	...	6·6 ohms resistance
B_3	...	100 v. high-tension battery
L	...	15 w., 210 v.
MR	...	Westinghouse J 50
$P_1 = P_2$	...	Osram KMV 6 (potassium thin-film)
T_1	...	250–0–250 v. A.C.
T_2	...	50–0–200 v. A.C.

FIG. 6

3. AMPLIFIER CIRCUIT

Each photocell is provided with its own independent D.C. amplifier, and the outputs from the two are finally combined in series opposition, as already described. The amplifiers used must obviously be very stable, and since the stability of conventional types of D.C. amplifiers, particularly if mains-driven, is generally poor, a special type was developed for the present case. The circuit used is a modified version of one described by Walker[2] and attributed in the first place to the Radio Corpn. of America. The full circuit details are shown in Fig. 6. It is a symmetrical arrangement of two identical amplifiers, powered by a two-phase alternating current supply derived from the centre-tapped transformer secondary T_1, of 250–0–250 v. The valves V_1, V_3, and V_5 constitute one amplifier, serving the photocell P_1, and V_2, V_4, and V_6 the other, serving the photocell P_2. The valve V_7 is a gas-filled relay to which the combined outputs of the two amplifiers, which appear as D.C. voltages with the polarity shown across resistances R_{19}, are applied. The operation of the circuit is as follows—

During that half-cycle of the A.C. supply which renders the upper end of the secondary of T_1 positive to the centre-tap (referred to as the "positive half-cycle"), the anode of V_1 is positive with regard to its cathode, and the valve therefore passes a pulse of current through the resistance R_{15}, and thereby causes a potential difference to appear across this resistance which charges the condenser C_3 with the polarity shown. The magnitude of the charge so imparted to C_3 will depend on the average potential of the control grid of V_1, which is determined by the intensity of light falling on P_1, as will be described later (p. 183). During the succeeding or "negative" half-cycle, V_1 cannot conduct, but owing to the fact that the time-constant of R_{15} and C_3 is long (0·5 sec.) compared with the period of the A.C. supply (0·02 sec.), C_3 will not appreciably discharge before the following positive half-cycle again tends to charge it. The valve V_1 thus behaves as a half-wave rectifier, and C_3 charges to a potential difference approximately equal to the peak value of the voltage drop down R_{15} on positive half-cycles. This value would, in absence of other circumstances, be approx. 125 v., half the applied anode voltage, and can be adjusted by varying the screen-grid potential of V_1 by means of the potentiometer R_8. During negative half-cycles, however, when V_1 is not conducting, a pulse of current flows through R_{15} through the metal rectifier MR_1, connected the reverse way round to V_1, thus tending to discharge C_3. Ignoring for the moment the bias voltage introduced by battery B_3, whose purpose will be described later, and assuming both rectifiers MR_1 and MR_1' to be returned directly to earth and not to the battery, it is plain that the extent to which C_3 is discharged during half-cycles is determined by the value of resistance R_{16}, and can in consequence be adjusted so that, with V_1 removed, C_3 would charge to approx. 120 v. in the opposite sense to that shown. Thus, with both V_1 and MR_1 in operation, there is a small net charge on C_3, of the polarity shown, of 4–5 v., and this value is readily increased or decreased by adjustment of R_8 or R_{16}. Since the latter is not altered after the initial setting, once R_8 is set any change in the potential at the control grid of V_1 will appear in amplified form as a change in the charge on C_3: V_1 thus acts as a D.C. amplifying stage. Moreover, the small residual charge on C_3 is of the right magnitude and polarity to apply as control grid bias directly to the valve V_3, connected "back to back" with V_1, as shown, across the same A.C. supply. This valve also has a metal rectifier MR_2 to balance off part of the voltage drop normally occurring down R_{17}. In this case the rectifier is arranged, by adjustment of the values of R_{17} and R_{18}, to provide a voltage across R_{17} in excess of that set up by V_3, so that the net charge on C_4 has the polarity shown, and a value of 40–60 volts. Thus, V_3 provides a second stage of D.C. amplification in cascade with the first. Finally, the voltage across C_4 is applied to the grid of V_5, which is connected as a cathode follower to provide a low-impedance output for application to the meter V and the relay stage V_7. It will be plain that the other amplifier V_2–V_4–V_6 functions in precisely the same manner on alternate half-cycles of the A.C. supply, and provides an output across C_5' which can be made to balance exactly that derived from the upper section. This balance adjustment is most conveniently made by means of R_6, which is adjusted until the centre-reading voltmeter V indicates zero voltage across the two output condensers C_5 in series.

The gas-filled relay V_7 is supplied with A.C. anode voltage from a separate transformer secondary T_2 (50–0–200 v.), and the voltage across the 50-v. (lower) section is rectified by means of the diode V_8, and smoothed by C_8, C_7, and R_{23}, before being applied as negative D.C. bias, adjustable by means of the potentiometer R_{20}, to the grid of V_7 to restrain the discharge of the latter. The net output from V_5 and V_6 is connected in series with this bias voltage, so that V_7 conducts when the net output exceeds a value of approx. 1 v., the exact figure being adjustable by means of R_{20}. When V_7 conducts, the relay $A/1,2$ in its anode circuit closes, and the contact $A/2$ operates one or other of the automatic burette valves B_1 and B_2, according to the position of the switch S_2.

One difficulty was encountered in the operation of V_7: this valve draws a rather heavy anode current to operate the relay, and the current is half-wave rectified, i.e. it coincides with the positive half-cycle of voltage in the main amplifier, but not with the negative half-cycle. Because both V_7 and the main amplifier are supplied from the same transformer, and the regulation of such small transformers is not perfect, the firing of V_7 causes the voltage of the positive half-cycle in the main amplifier to decrease slightly, while that of the negative half-cycle remains unaffected. Thus, the voltage delivered to C_3 by V_1 is decreased, whereas that delivered by MR_1 remains unchanged; on the other side, the voltage delivered to C_3' by V_2 is unaffected, whereas that from MR_1' is decreased. The amplifier is thus unbalanced, and the out-of-balance voltage tends to "lock in" the gas-filled relay and reduce the sensitivity of triggering. The effect is overcome by causing a second contact $A/1$ of the relay to switch in, when V_7 conducts, a dummy load (R_{21} and R_{22}) across the phase not affected by thyratron current. The current drawn by this dummy load can be adjusted by means of R_{22} so that it exactly compensates the out-of-balance effect, and the main amplifier remains unaffected when V_7 fires. If the system is slightly overcompensated by reducing R_{22} below the exact compensation value, it is found that the operation of V_7 becomes intermittent over a certain range with a period of about 1 sec., determined mainly by the time-constant of the transformer secondary. This phenomenon is useful, because it enables an approximation to proportional control to be obtained. Thus, as the output from the amplifiers increases, the thyratron conducts at 1 v. output, but at first only intermittently, and the burette is turned on and off for approximately equal time periods, i.e. it delivers at about half the rate corresponding to continuous operation; when the output from the amplifiers exceeds 5 v. (i.e. when fairly considerable correction is required in the test solution), however,

the intermittency ceases and the burette delivers continuously, thus doubling its rate of delivery. As the output from the amplifier decreases owing to the resultant addition of dye to the test solution, the operation again becomes intermittent at 5 v., and ceases at 1 v. Normally, the 5-v. output is attained only at the beginning of a run, when absorption of dye from the test solution is very rapid, particularly at temperatures of 60°C. and above.

4. AMPLIFIER STABILITY

Theoretically, amplifiers of this type should be insensitive to mains voltage variations, since they operate on alternate half-cycles of the same supply. Thus, if the applied voltage falls, the current drawn by V_1 during positive half-cycles decreases, but the current drawn by the metal rectifier MR_1 during negative half-cycles also decreases, leaving the charge on C_3 relatively unaffected. Such compensation can be only partial, however, since V_1 and MR_1, though they both function as half-wave rectifiers, do not possess the same type of voltage–current characteristic. Thus, in Fig. 7 the straight

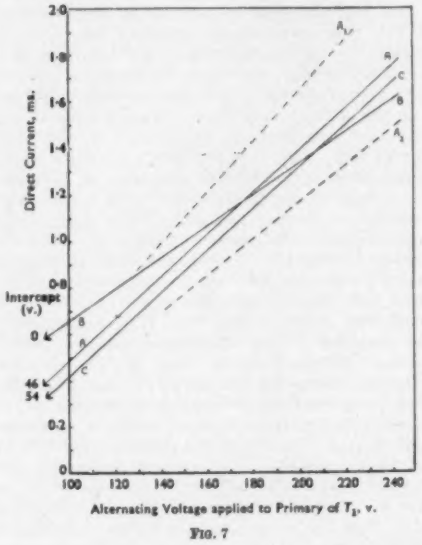


FIG. 7

line A shows the current flowing in the anode circuit of V_1, as a function of the A.C. voltage applied to the *primary* of transformer T_1. The current values were obtained by breaking the circuit at the anode of V_1, and inserting a milliammeter. Normally, the anode current of a pentode valve is independent of the applied voltage if the latter is above the "knee" of the characteristic curve; but such characteristics are taken with the control and screen grid voltages and the cathode temperature held constant. In the present case, when the voltage applied to T_1 is varied, all three vary together with the anode voltage, with the result shown, namely that the anode current is a linear function of the applied (mains) voltage over the range studied, and the line makes an apparent intercept on the voltage axis of approx. 46 v. By varying the screen voltage, the gradient of the line can be varied between approximately the limits shown at A_1 and A_2, the intercept on the voltage axis remaining constant.

If, on the other hand, the milliammeter is inserted in series with the rectifier MR_1, the line B is obtained; again the current is a linear function of the mains voltage applied to T_1, but the line passes very approximately through the origin. Its gradient can be altered at will by varying the series resistance R_{16}. Now, the charge on the condenser C_3 at any instant is proportional to the difference between these two currents, and will for example be zero for an applied voltage of 177 v. For values greater than 177 v. the condenser charge will have the polarity shown in Fig. 6, and for values less than 177 v. the polarity of the charge will reverse. If the amplifier is to be stable towards variations in mains voltage, however, it is essential that the charge on C_3 shall remain constant despite such variations. This can be effected by altering the shape of the voltage–current characteristic of the metal rectifier MR_1 until it lies parallel with, and slightly below, that of the valve over the whole of the mains voltage range likely to be encountered in practice. The difference in currents is then constant, and the charge on C_3 cannot alter, no matter how much the mains voltage may vary within this range. The necessary alteration in the shape of the rectifier characteristic is brought about by inserting in series with the rectifier a D.C. bias voltage derived from the battery B_3 with the polarity shown. This gives the characteristic an intercept on the voltage axis whose value can be adjusted at will; and once the correct intercept has been found, the slope of the characteristic can be brought to the correct value by adjustment of R_{16}. Line C in Fig. 7 shows the form of the adjusted characteristic, with 63 v. D.C. bias in the rectifier circuit. As a result of the adjustment, the charge on C_3 was found not to alter when the mains voltage applied to T_1 was varied from 160 to 240 v. R.M.S.

If the whole amplifier is to be stabilised in this way, it would appear to be necessary to repeat the above procedure for the second stage (V_3) and its associated rectifier, and, of course, for both stages of the other amplifier (V_2 and V_4). In practice, however, it is found that stabilisation of the second stage is not necessary, as both first and second stages can be stabilised simultaneously by suitably overcorrecting in the first stage and leaving the second stage uncorrected. Following this procedure, the amplifier was found to be capable of almost perfect stabilisation against mains voltage variations, so much so that on varying the applied mains voltage from 160 to 240 v., the output of the balanced amplifiers as measured from the cathode of V_5 to the cathode of V_6 did not depart from zero by more than 0·5 v. Undoubtedly, one reason why such perfect stabilisation is achieved is that the method used takes into account and allows for the variations in cathode emission which occur when mains voltages change, if the valve heaters are run

from windings on the mains transformer. Most stabilising arrangements ignore this factor, and are imperfect in consequence*.

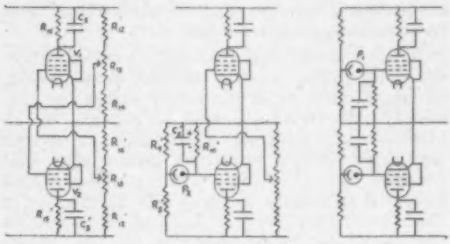
While the amplifier itself is thus rendered very stable, there remains the question of a stable light source. Again, the instrument should theoretically be self-compensating in this respect, since a double-sided circuit involving two photocells is used; but commercial cells are never so closely identical in characteristics as to make entire reliance on such compensation possible. In practice it is found that the use of a double-sided circuit reduces the errors caused by varying lamp voltage to about one-tenth of what would be found if the circuit were single-sided. This, however, is not always sufficient, and in consequence, in the present case, since the lamp derives its supply from a winding on the mains transformer, the whole instrument was supplied from a constant-voltage transformer (230 v., 150 w., Advance Components Ltd.). This was found to be necessary only when the instrument was used photoelectrically. When used for conductimetric work (see below), it was supplied direct from the mains.

5. PHOTOCELL INPUT CIRCUITS AND STANDARDISATION PROCEDURE

The input grid circuits of valves V_1 and V_2, involving the two photocells, are so arranged that, after standardising at the start of a run, the same amount of light (measured in an electrical sense, by the output it produces from the photocell) always falls on the cell cathode, irrespective of the concentration or colour of the dye solution in use. This being the case, it is plain that a given variation in this amount of light will always produce the same variation in voltage at the grid of V_1 or V_2, and hence the same output applied to V_7. The sensitivity of the apparatus is thus constant for all conditions of dye concentration and colour, in the sense that a given percentage difference in the light transmitted by the standard and test solutions will always cause the burettes to operate, no matter what test solution is in use. This percentage difference, which may be termed the "transmission sensitivity", is approx. 0·25%, as will be shown later. The change in dye concentration necessary to cause this change in transmission is not constant, but depends on the initial concentration, being greatest in the most concentrated solutions. Thus the "concentration sensitivity" is greatest in dilute solutions, and least in very concentrated ones, but is adequate to maintain equality between the concentrations of standard and test solutions in all cases so far investigated. Where deviations have been observed, as described later, they have in all cases been caused by factors extraneous to the electrical and optical system.

The method used to ensure this constancy of light intensity at the cathodes of the photocells under all circumstances is as follows—

The grid circuits of both V_1 and V_2 are switched by means of a quadruple 6-point rotary switch, all four wafers being ganged on the same shaft, as shown in Fig. 6. The six positions are divided into two groups of three positions each, one group being used for white (i.e. unfiltered) light, and the other group when colour filters are used, as indicated in connection with the second wafer from the top in Fig. 6. In order to simplify description, the circuits set up in each of the three switch positions of one group have been extracted and are shown in Fig. 8 A, B, and C. Thus, with all wafers in position 1 on the "white light" side, the circuit established is that shown in Fig. 8A.



(A) (B) (C)

FIG. 8

Here, the control grid of V_1 is connected to a point on the resistance R_{13}', which forms part of a potentiometer chain connected across the opposite phase. Thus, as the anode of V_1 swings positive, the grid of V_1 must swing negative, so that R_{13}' provides in effect a standard negative grid bias for V_1; similarly R_{13} provides grid bias for V_2. These bias voltages are preset to 1·5 v. R.M.S., and are not thereafter altered. The first step in standardising is, therefore, to switch to the first position, and balance the amplifier output to zero by means of R_6 (Fig. 6).

The rotary switch is then placed in position 2, whereby the circuit is altered to that of Fig 8B. The valve V_1 retains its standard bias, but the photocell P_2, with its load resistance R_{10}' and condenser C_2', is connected to the grid of V_2, as shown. It will be plain that when V_2 is conducting, the photocell is not, since its cathode is positive to its anode by reason of the potential difference across R_4'; but on the succeeding half-cycle, when V_2 does not conduct, the cathode of P_2 swings negative to its anode and a pulse of current flows through R_{10}', charging the condenser C_2' with polarity shown. Again, a long time-constant prevents C_2' from discharging appreciably during the idle half-cycle, so that the grid of V_2 is maintained negative to an extent depending on the intensity of light falling on the cathode of P_2. This is adjusted by means of the lamphouse slides, with the internal lamps in the photocell housings as a fine adjustment, until the amplifier output is again zero. Then, since the amplifier was initially balanced with an R.M.S. (A.C.) voltage of 1·5 v. on this grid, the D.C. voltage across C_2 in the new balance must have a definite value equivalent in its effect on V_2 to the 1·5 v. which it replaces. The value will not

* A rather full account of the amplifier circuit and its stabilising arrangements has been given, because it is felt that these constitute a new approach to the problem of stable D.C. amplification. Such amplifiers are increasingly in demand for use in connection with electronic measurements, and the circuit may well have wider applications than the particular one involved in the present case.

be exactly 1·5 v., but will approximate very closely to this figure, and will always be the same.

Having thus standardised P_2, the rotary switch is moved to its third position, which establishes the final circuit (Fig. 8c), bringing the other photocell P_1 into the grid circuit of V_1 in a precisely similar manner. Without altering the adjustments of P_2, the amount of light falling on P_1 is now varied until the output of the amplifier is again zero. Then, both grids must be at a standard potential of approx. — 1·5 v. (relative to cathode), and the standardising routine will ensure that this figure is always maintained, irrespective of what solutions the light may be passing through, provided always that sufficient light can reach the photocell cathodes to give the necessary output.

If very concentrated dye solutions or deep filters are in use, this may not be the case; consequently, the three positions of the rotary switch not previously referred to are used to provide, by an identical standardising procedure, an overall sensitivity to light five times greater than that obtained from the "white light" positions. This increased sensitivity is simply due to the use, in the "filter" positions, of higher load resistances for the photocells, those employed for the "white light" side being 30 megohms and those for the "filter" side 150 megohms. Whichever sensitivity is used, the intensity of light falling on the photocells is adjusted to provide a potential of 1·5 v. across the load resistance. It is obvious that much less light will be necessary with the greater load resistance, and hence it is possible in this case to work with much more concentrated solutions and deeper filters than is feasible with lower load resistances. The operating sensitivity, or transmission sensitivity already referred to, is not affected.

The transmission sensitivity may readily be calculated. Since the amplifier requires 1 v. output to operate the relay, and the gain of the amplifier is 300, the change in voltage at the first valve grid corresponding to 1 v. output is approx. 3·3 mv.; since light electrically equivalent to 1500 mv. is falling on the photocell, the percentage change in this light necessary to cause 3·3 mv. difference at the grid is approx. 0·25%. Hence, the relay system operates for a change of 0·25% in total light transmitted by the test solution.

6. ALTERNATIVE CONTROL BY CONDUCTIVITY

Experience in the photoelectric control of dyeing, as described above, soon showed that disturbing factors not hitherto recognised were present, which tended to cause the actual dye concentration in the test solution to diverge from that of the standard, *even though the total light transmission through the two solutions remained identical.* The difficulty will be discussed at greater length at a later stage, but it occurs chiefly at low pH values (2–3) in solutions containing only free dye acids, such as Orange II. The effect was finally found to be due to the appreciable amount of wool substance which passes into solution in these circumstances; the accumulation of colloidal hydrolysis products in the solution results in a considerable amount of light absorption by scattering. When this occurs, the photometer automatically allows the dye concentration to fall, so as to maintain constant transmission. Under these circumstances, and providing the dye itself is the only source of ions in the solution, closer control can be obtained by modifying the arrangements so as to keep the electrical conductivity of the test solution, rather than its light transmission, constant throughout the experiment; the wool hydrolysis products appear to be too colloidal to contribute appreciably to the total conductivity, and in any case are probably amphoteric in character, or largely so.

The necessary modifications to the circuit are embodied in Fig. 6. The rotary switch is turned to position 1, and the amplifier balanced as already described; then the switch S_1 is moved from the "photocell" to the "conductivity" position. The pair of electrodes immersed in the test solution, already referred to (p. 178), are connected to the terminals E, and are thus placed in series with an adjustable source of alternating voltage R_2 and a load resistance R_{11}, the latter being connected between the grid and cathode of V_2. The voltage across this load, which now replaces the standard bias from R_{13}', can be set to 1·5 v. by adjustment of R_2; the amplifier is thus rebalanced, and any variation in the conductivity of the test solution will cause the voltage across R_{11} to vary, and thus operate the relay system. The voltage available across R_2 is approx. 60 v., and with very dilute solutions this may be insufficient to allow 1·5 v. to be developed across R_{11}. Rather than increase the voltage, alternative pairs of electrodes are available of different superficial area. In practice two pairs are sufficient to cover the pH range 1–4. For the lower values, the exposed platinum surface of the electrodes consists of about 0·5 mm. of 22 s.w.g. platinum wire, and for the higher ones, about 5 mm. of the same wire, coiled into a close spiral, is used. Above pH 3·5 photoelectric control is preferable, since the conductivity of the test solution then becomes extremely low. Within the range mentioned, one or other of the pairs of electrodes will give a solution resistance lying between 5000 and 30,000 ohms, a range which can be accommodated by the voltage available at R_2. The electrodes are covered with platinum black by the usual electrolytic methods.

7. GENERAL PROCEDURE IN USING THE PHOTOMETER

The following routine has been found suitable when using the photometer for dyeing rate determinations—

The U-tube was thoroughly rinsed with the dye solution to be used, 200 c.c. of the solution placed in it, and the tube clamped in position in the thermostat tank, which was then brought up to the temperature required. The standard cell was also filled with the dye solution, covered with its metal cap, and placed in position in the light-beam. Air bubbles were cleared from the inner faces of the inspection cell of the U-tube by a few rapid strokes of the piston; formation of air bubbles on the external surfaces was prevented by wiping the

glass externally with a tuft of cotton-wool moistened with alcohol before each experiment. The piston was started, and when the motor driving it had reached a speed of 38–40 strokes per minute, the amplifier was balanced and standardisation carried out as previously described. If photoelectric control was to be used, the pump was stopped before obtaining a balance, as it was found that, in the absence of the wool, the pump action was sufficiently violent to cause a stream of air bubbles to form in the inspection cell, leading to a false standardisation. If the conductimetric circuit was to be used, however, a stationary liquor caused local temperature effects in the region of the electrodes, and a balance was therefore obtained with the pump in operation. After standardising, the dye solution was circulated for a further 15 min. in order to make sure that temperature equilibrium had been reached, the light source was sufficiently steady, and a true zero had been obtained. (At times, despite the constant-voltage transformer, fluctuations in mains voltage were such as to cause sufficient error to vitiate results. Such variations occurred chiefly round about 12 noon and 5.15 p.m., and by avoiding these times, few experiments were wasted. Variations of this character were easily detected during the course of an experiment by plotting the rate curve as the readings were taken. They did not occur at all when conductimetric control was used, indicating quite definitely that their origin lay in the effect of the varying mains voltage on the light source.)

The weighed wool, loosely wrapped round a glass spreader, was first wetted out in boiling distilled water for 15 min., shaken quickly to remove surplus water, and then plunged into the dye solution, the timing of the experiment being taken from the moment of complete immersion of the material. The thermometer was replaced, the pump restarted, the burettes placed in position, and the burette relay circuit switched on. The time of the complete operation from the entry of the material to the bringing into operation of the burettes was 5–10 sec. Readings of the burettes were taken at intervals which varied with the conditions of the experiment. Normally, the intervals were 1 min. for the first 10 min., 2 min. up to 20 min., and then 5, 10, and 30 min.

If necessary, towards the end of the experiment, when the rate of addition of dye had become very small, the alternative burette was brought into use. Two burettes were fitted, one of 10 c.c. capacity graduated to 0·1 c.c., the other of 2 c.c. graduated to 0·01 c.c. Either could be brought into operation by means of the two-way switch S_2 (Fig. 6). In this way dye solution could be added rapidly at the beginning of an experiment, when the rate of absorption was large, from the large burette, and slowly towards the end from the small burette, with equal accuracy in reading. If necessary, a more dilute dye solution could be used in the smaller burette, so as to increase still more the accuracy of reading towards the end of an experiment, or during experiments at low temperatures.

At the end of an experiment the burettes were removed from position immediately after an addition had been made to the dye solution. As soon as balance had once more been established, the thermometer was removed, and the action of the pump then brought the material to the top of the U-tube, from which it could be rapidly removed with the assistance of a hooked glass rod. After switching off burette and test circuits, the apparatus was removed from the tank, and the residual dye liquor decanted into a glass-stoppered flask and cooled to 18°C. The pH was then determined.

8. ACCURACY AND REPRODUCIBILITY OF RESULTS

The experimental results which follow are given solely for the purpose of illustrating the degree of accuracy and reproducibility obtainable with the instrument described. The dye used throughout was a specially purified sample of Orange II (free acid), and the wool used was from a New Zealand Romney fleece (shoulder and back). Approximately one-third of each staple was cut from the tip ends and rejected. The remainder was extracted in a Soxhlet apparatus for 24 hr. each with ether and alcohol, and washed in distilled water. After squeezing off, the material was immersed in 0·001 N. hydrochloric acid for 24 hr., and then successively steeped in distilled water until an equilibrium pH of 4·8 was established. It was then hydroextracted, pressed between filter papers, and stored in a room at 65% R.H. and 22·2°C. until required.

(*a*) *Inaccuracy of Photometric Control at Low pH Values*

It has already been indicated that at pH values lower than about 3·5 photometric control is inadequate to maintain constant dyestuff concentration in the test solution. The following experiment illustrates this point—

A solution (200 c.c.) of the free acid of Orange II of pH 3·18 was introduced into the apparatus and brought to 60°C. The wool sample (2·2 g. conditioned weight) was then entered, after balancing and standardising the photometer, and the burettes and relays were switched on. The burettes contained an approx. 0·02 N. solution of Orange II free acid. Dyeing was continued for 3 hr., during which time a zero reading was maintained on the output voltmeter by automatic additions from the burettes as dye was absorbed by the wool, and at the end of the experiment balance was still preserved, apparently indicating that the concentration of the test solution had remained unaltered.

After removal of the wool, the contents of the U-tube were cooled to 18°C. and the pH of the solution was measured. It was found to have increased to 3·64, and visual comparison with the original solution showed the dye concentration to have decreased considerably. Further confirmation was obtained by titrating aliquots of the original and final solutions with titanous sulphate, with the following results—

20 c.c. of original solution required 7·1 c.c. of titanous sulphate
20 c.c. of final solution required 2·9 c.c. of titanous sulphate
(Ammonia content of final solution = 0·2 milliequiv. per litre)

Since these titration values show that the dye concentration of the final solution is 40·7% of that of the original, the pH of the final solution should be 3·57. The difference between this and the value found (3·64) is accounted for by the ammonia liberated by hydrolysis of amide side-chains.

Examination of the original and final dye solutions in the Spekker absorptiometer confirmed that the light transmissions of the two were in fact equal. It therefore seemed most probable that the used dye solution contained colloidal particles capable of scattering a proportion of the incident light, and examination of the solution in an intense focussed light beam against a black velvet background revealed the existence of a pronounced Tyndall effect. Meggy[3] has since reported that solutions of the free acid of Orange II which have been in contact with wool for some time deposit, on standing, an insoluble product said to consist of a compound of dye and wool. As, however, the data given above indicate that the hydrogen ion concentration of the final solution is completely accounted for in terms of the anion concentration and the concentration of ammonia present, it follows that whatever substance is responsible for the dispersion of the light is not influencing the pH value of the solution. In this fact lies the justification for the use of conductimetric control along the lines already indicated.

When the above experiment was repeated with a more dilute dye solution having a pH of 3·8, the final solution was found by titanous sulphate titration to have a dye concentration equal to that of the original, and the pH values of the two were identical, indicating that hydrolysis, in so far as it affects accurate photometry, is negligible at this pH.

Finally, a series of experiments were run to determine whether conductimetric control could be used at the lower pH values. The conditions were identical with those already given, the dye solution having a concentration of approx. 0·001 N. and an initial pH of 3·00. Dyeing was carried out for 3 hr. at a series of temperatures, and the pH and ammonia content of the final solution were determined. Conductimetric control was used in all cases, and the results shown in Table I were obtained.

TABLE I

(Initial pH = 3·00)

Temperature (°C.)	Final pH	Ammonia Content (milliequiv. per litre)	pH Equivalent of Ammonia Content
40	3·02	0·04	0·02
50	3·05	0·11	0·04
60	3·09	0·19	0·07
80	3·15	0·30	0·15

Aliquots of the initial and final solutions were titrated with titanous sulphate, and the chromophore content was found to be maintained; the hydrogen ion concentration, however, decreases by an amount accounted for by the amide hydrolysis, and it is not possible to avoid this slight change in pH when using conductimetric control.

(*b*) *Comparison of Conductimetric and Photometric Control*

In the region of pH 3·5 it is possible to use either method of control, and experiments were therefore performed to effect a comparison of the two methods under otherwise identical conditions. Dyeing was carried out at 60°C., using firstly the photometric, and secondly the conductimetric method. The results of two typical experiments are shown in Tables II and III.

TABLE II

Temperature	60°C.
Dry wt. of wool	1·91 g.
Volume of solution	200 c.c.
Concn. of soln. in burette ...	0·02201 N.
Initial pH	3·50
Final pH	3·55

Time (min.)	Addition (c.c.) Photometric	Conductimetric
1	—	1·77
2	3·00	2·72
3	3·80	3·62
4	4·55	4·35
5	5·25	5·10
6	5·85	5·75
7	6·45	6·40
8	7·00	6·96
10	8·00	8·02
12	8·95	9·02
14	9·80	9·92
16	10·60	10·75
18	11·37	11·50
20	12·10	12·28
25	13·77	13·97
30	15·25	—
35	16·60	16·70
40	17·85	17·92
45	19·02	19·07
50	20·02	20·05
60	22·00	22·10
70	23·72	23·80
80	25·32	25·41
90	26·72	26·81
100	28·00	28·05
120	30·30	30·32
140	32·20	32·20
160	33·12	33·10

These data indicate that, although the amount of dye added in the same time by the two methods may differ by 10–15% of the total dye added during the first 5 min. of the experiment, the variation is within approx. 1% after that period. The higher figure for the early period is probably due to small errors in timing the beginning of the experiment. The differences in the later stage are so small that they lead to identical values for the slope of the linear graph obtained by plotting the amount of dye absorbed against the square root of the time.

(*c*) *Effect of Rate of Circulation of Liquor on Rate of Absorption of Dye*

Although the motor operating the circulating piston runs at constant speed, it was possible to vary the volume of liquor passing through the wool sample in unit time, by altering the stroke of the piston. This was effected by altering the throw of the eccentric driving the piston, several alternative

attachment points being provided for the driving crank. Using the shortest piston stroke, approx. 750 c.c. of liquid was displaced per minute, i.e. the whole of the dye solution passed through the sample

TABLE III

Temperature	60°c.
Dry wt. of wool	1·91 g.
Volume of solution	200 c.c.
Concn. of soln. in burette ...	0·02064 N.
Initial pH	3·67
Final pH	3·57

Time (min.)	Addition (c.c.) Photoelectric	Conductimetric
1	1·17	1·21
2	2·07	2·10
3	2·85	2·90
4	3·55	3·61
5	4·17	4·17
6	4·73	4·75
7	5·32	5·40
8	5·77	5·75
9	6·27	6·31
10	6·73	6·75
12	7·57	7·59
14	8·36	8·39
16	9·12	9·10
18	9·80	9·82
20	10·47	10·48
25	11·98	11·98
30	13·38	13·41
35	14·63	14·65
40	15·80	15·85
45	16·87	16·90
60	19·66	19·68
70	21·10	21·15
80	23·40	23·40
120	25·85	25·88
150	27·42	27·50

3½ times per minute; whereas the longest piston stroke gave a displacement of 1500 c.c. per minute, and the whole dyebath passed through the sample 7½ times per minute. In order to determine whether variations in the rate of circulation were likely to affect the rate of absorption of dye by the wool, experiments were performed, under identical conditions of dye acid concentration and temperature, using the maximum and minimum rates of circulation respectively, with the results shown in Table IV.

TABLE IV

Dry wt. of wool	1·91 g.
Volume of solution	200 c.c.
Temperature	60°c.
Concn. of soln. in burette ...	0·0218 N.
Final pH value	4·00

Time (min.)	Addition (c.c.) 750 c.c./min. Circulation	1500 c.c./min. Circulation
1	0·88	0·75
2	1·45	1·23
3	1·89	1·65
4	2·30	2·07
5	2·70	2·43
6	3·07	2·97
7	3·45	3·58
9	4·06	—
12	4·91	4·86
14	5·47	5·32
16	5·97	5·72
18	6·45	6·15
20	6·92	7·10
22	7·30	7·45
25	7·87	7·83
30	8·81	8·73
35	9·65	9·43
40	10·42	10·10
50	11·77	11·87
60	13·05	12·92
70	13·75	13·58
80	14·07	—
90	14·80	14·63
100	15·31	15·00

Thus, it is plain that small variations in the rate of circulation are without effect on the rate of absorption of dye under the experimental conditions employed.

DEPARTMENT OF TEXTILE INDUSTRIES
THE UNIVERSITY
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(*Received 18th December 1950*)

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PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—I

Development of the Society's Light Fastness Standards (B.S.1006)

FASTNESS TESTS CO-ORDINATING COMMITTEE

During the course of the last few years there have appeared in the Continental press a number of statements which indicate that the facts relating to the development of light standards in Great Britain are not fully known. The general tenor of these statements is indicated by the following extracts—

(i) . . . . . the German standards with two unimportant exceptions are used in England to-day (P. RABE [1]).

(ii) . . . . . England have in essentials adopted the blue scale on wool worked out by the old D.E.K., with certain minor modifications (W. WELTZIEN [2]).

(iii) The blue standards established by the D.E.K. in 1936 for the assessment of light fastness have now been reproduced in England . . . . . (C. ZUBER [3]).

(iv) . . . . . the eight-step blue scale on wool, which was first published in the seventh edition of the D.E.K. test methods in 1935 (H. RIS [4]).

The Fastness Tests Co-ordinating Committee would therefore like to draw attention to the history of the development of these standards in Great Britain.

The work carried out by the Society on fastness tests was started in 1928, when a small number of investigators were appointed and arrangements were made for practical work to be carried out in the laboratories of the British Research Association for the Woollen and Worsted Industries (now the Wool Industries Research Association). An announcement to this effect was made in July 1928 [5].

By July 1929 it was possible to report [6] on the results of a large number of fading tests which had been carried out on fabrics composed of different fibres and in different parts of the world. The influence of humidity on the degree of fading had been investigated in the laboratory and was discussed in some detail in this paper. It was stated that an extensive examination of the existing German light fastness standards on wool, cotton, and silk was being undertaken.

In an article in 1930 [7], Dr. P. Krais, then Director of the German Textile Research Institute in Dresden, discussed various aspects of fading. After referring to the differences between the results obtained with fading lamps and sunlight, the author proceeded to discuss "the practical method", by which he referred to the use of dyed standards.

At this time the Deutsche Echtheitskommission (D.E.K.) had separate light fastness standards for each of the following fibres—(*a*) cotton, (*b*) wool, (*c*) unweighted silk, (*d*) weighted silk, (*e*) acetate "silk", and (*f*) viscose "silk"—i.e. 48 separate dyeings were used. Moreover, with any one fibre the eight standards were dyed in different colours; for example, the wool standards comprised red, green, blue, and violet dyeings.

The remarks of the author of this paper (Dr. Krais) make interesting reading—

> A second point of doubt is the choice of standard dyeings the German Commission [D.E.K.] has come to. I am sure the English and American Commissions think to be able to improve on this. No doubt they will be able to do so, but what will it avail? These standards are chosen with the utmost consideration and on the basis of many years' experience. And with these very standards we have now had sixteen years' experience on the top of that, in any way with those on cotton and wool, and we are quite satisfied with them as a working set, though they might not fulfil every ideal . . . . . Not a single discrepancy has crept up through all these years, though these standards are recognised by the Patent Office . . . . . The firms of the I.G. are using these standards now regularly. Would it, therefore, not be the best way, the simplest and quickest, if the commissions of the other countries would adopt them en bloc, as far as practical testing is concerned? . . . . .

On 7th April 1930, Dr. P. Krais met the Executive Committee of the Fastness Tests Committee and members of the British Research Association for the Woollen and Worsted Industries in Leeds to discuss international co-operation in fastness work [8]. He repeated the suggestion that the Society should accept the German standards as a whole, any alterations which might subsequently be found necessary being made by agreement.

The British Committee pointed out, however, that the German standards had been thoroughly examined by its research workers and had been found to have many defects and inconsistencies, details of which were given. The Committee was therefore unable to accept the German standards and intended to continue its investigations.

The Committee informed Dr. Krais, moreover, that, as a result of its work on this subject extending over two years, it had been found unnecessary to have standards for each test on separate fibres. In view of this it was intended to establish general standards on one fibre only. Dr. Krais said he would put before the German Committee the criticisms which had been made on the existing German standards and the suggestion that standards should be on one fibre only.

In a paper published in the *Journal* [9] during the same month as this meeting, it was stated that the considerable amount of data which had been obtained on fading led to the conclusion that the fading of dyeings on wool and silk was less affected by humidity than was the fading of dyeings on other fibres. The influence of humidity and of sulphur dioxide on 67 dyeings on different fibres was given in detail.

An "Interim Report October 1930" on the work carried out under the Society's fastness scheme was published in the *Journal* [10] in January 1931, describing briefly the results of the many exposures

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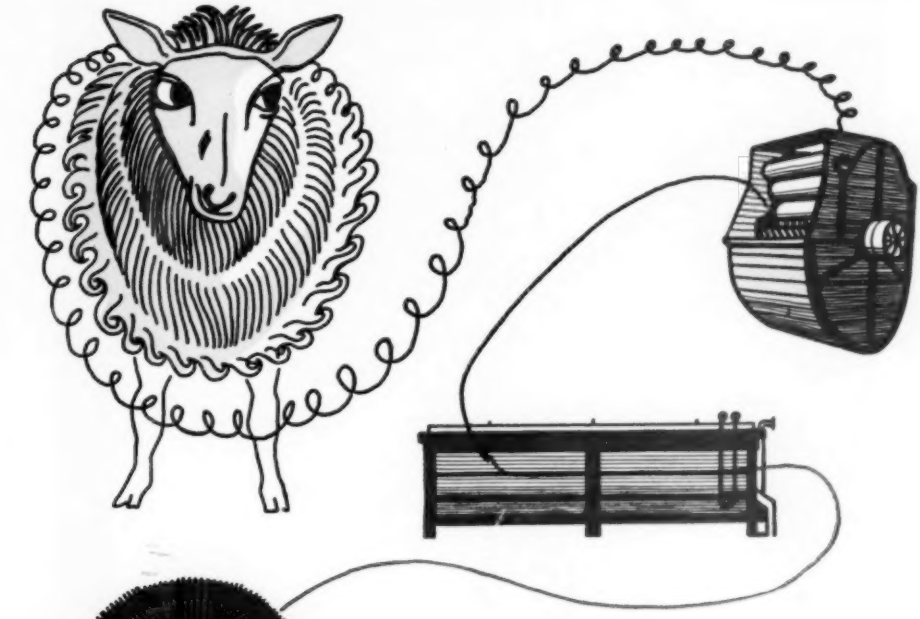

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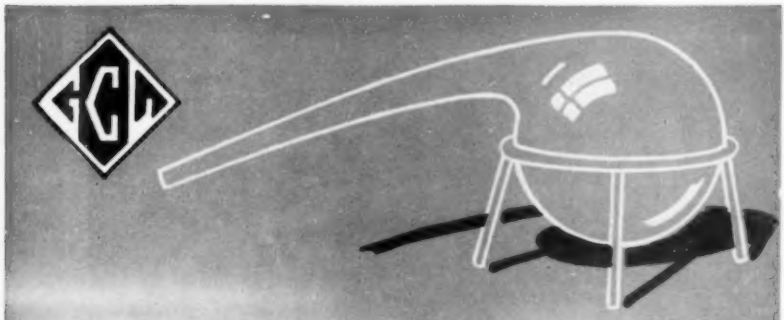

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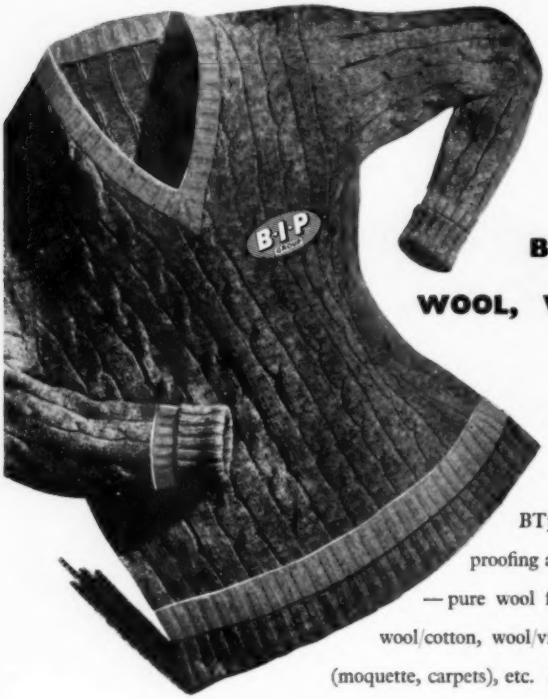

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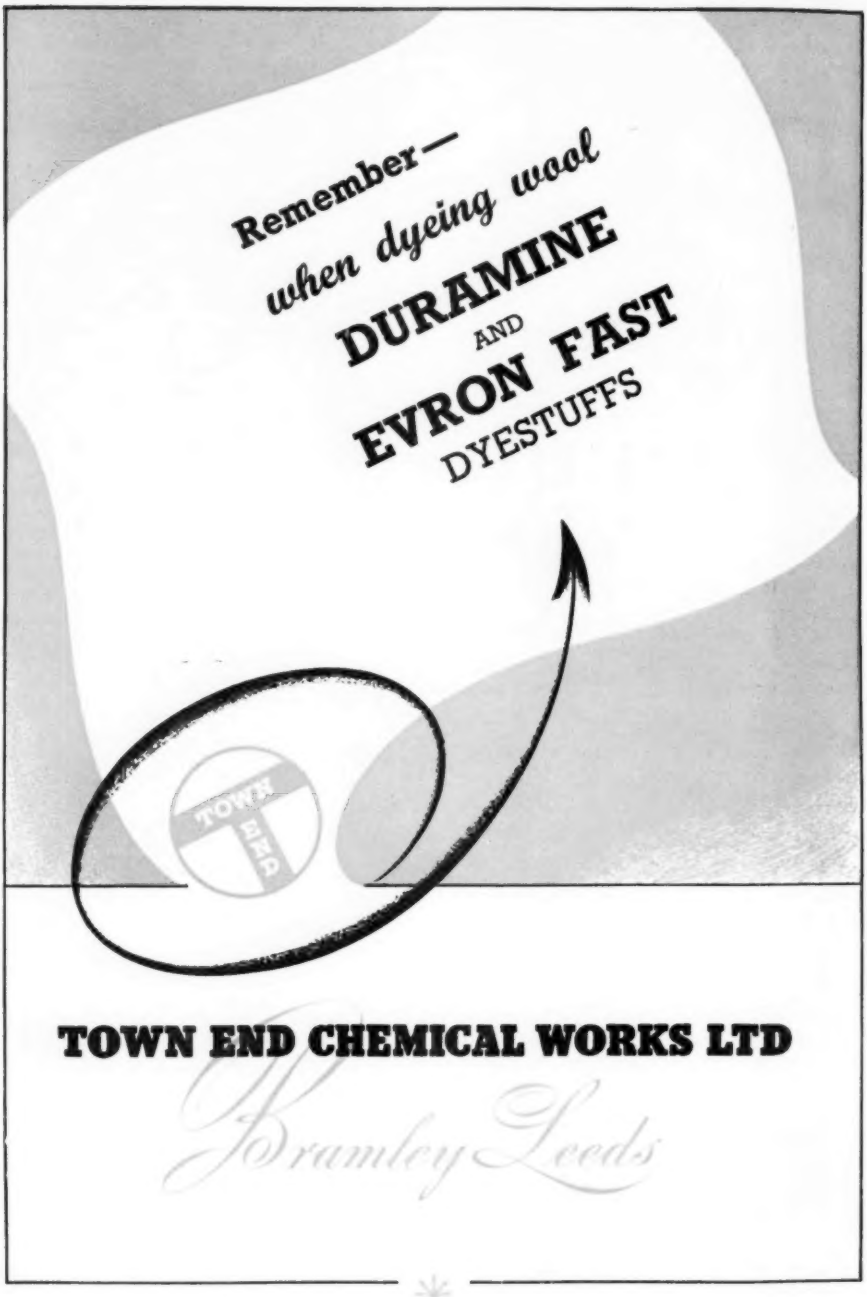
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carried out in 1928–1930; again discussing the influence of humidity, it was stated—

> The work of 1928 and 1929 having shown that silk and wool are the best fibres to use for the standard dyeings, the choice has fallen on wool, since silk has several disadvantages.

At this time it was intended to establish a range of light fastness standards in each of the colours red, green, and blue, on wool cloth, and a list was given of those dyes which had been found suitable for this purpose after considerable experimentation.

During the summers of 1931 and 1932 the gaps in the range of blue and red standards were filled, but the green range was abandoned. Owing to the approaching Jubilee of the Society, publication was delayed until 1934, when the "Report of the Society of Dyers & Colourists on the work of its Fastness Committee in fixing Standards for Light, Perspiration and Washing" was published. It may be noted that, in referring to the Interim Report of October 1930 with regard to the elimination of all fibres but wool, this report commented as follows—

> It is a significant fact that the proposals made at the time [i.e. 1930] by the Society's Fastness Committee have been subsequently used by prominent German and American organisations interested in the subject.

In 1932 there appeared a communication[11] from the laboratories of the I.G. Farbenindustrie A.-G. in which there were described the results of a large number of light exposures in eighteen different countries. It was stated that from these exposures they had selected a series of blue dyeings on wool which were to serve as the standards for all fibres. These standards were also given by the D.E.K. in the 6th edition of the "Verfahren, Normen und Typen" in 1932. It is clear, therefore, that the German and British standards were worked out simultaneously.

The standards as originally issued by the two committees were as follows—

Standard	German	British
1	0·6% Brilliant Wool Blue FFR extra	No standard
2	0·6% Wool Blue N extra	0·5% Lissamine Violet 6BNS
3	1·0% Brilliant Indocyanine 6B	0·6% Brilliant Indocyanine 6B
4	1·5% Wool Fast Blue GL	1·1% Polar Blue G conc.
5	1·3% Cyananthrol RX	0·5% Solway Blue RS (= Cyananthrol RX)
6	2·5% Alizarine Direct Blue AGG	2·0% Alizarine Light Blue 4GL
7	2·5% Indigosol AZG	4·0% Caledon Blue GCP 300 powder
8	3·0% Indigosol Blue AGG	3·0% Indigosol Blue AGG

Although there were differences in concentration, the similarity of the dyes chosen was quite remarkable.

During 1937–1939 a comparison was made of the light standards proposed by the German, American, and British committees. After protracted correspondence and meetings the three committees were almost agreed on a common set of standards. This was achieved largely through the fact that the American and British committees were prepared to give way on several standards in the general interest, although they were of opinion that there own standards were as good as those insisted upon by the D.E.K.

During the Spring of 1939 the negotiations had reached the stage when it had been agreed that an announcement would be made simultaneously in the three countries. In May 1939, however, Dr. Krais wrote a letter in identical terms to the American and British committees, making the following uncompromising statements—

(i) The scale of light standards was first worked out by the I.G., had been published in 1936, and was still in use.

(ii) The 8th edition of "Verfahren, Normen und Typen" was being prepared and would contain their original standards[12].

The D.E.K. also raised objections to the proposals for distributing the standards in America and Great Britain. The 8th edition of the tests was published shortly afterwards.

During July 1939 further correspondence took place and Dr. Krais submitted a draft announcement for publication. This contained statements which purported to give a history of the standards, but which were regarded by the Society as a claim that the establishment of the standards was due almost entirely to the I.G. Neither the Society nor the American Association of Textile Chemists and Colorists was able to accept this view, and they had reluctantly to conclude that agreement on a basis of equal partnership with the D.E.K. was not desired by the I.G. In September war broke out.

The original S.D.C. standards had been devised on the basis of medium depths, since these would introduce the least difficulty in comparing samples of any depth. During the 1937–1939 discussions, the standards were increased in depth in order to come into line with the D.E.K. On the failure of the discussions the Society retained the deeper dyeings in general, and in 1940 issued a revised set of standards[13], which in 1942 were adopted as national standards by the British Standards Institution[14].

The 1939 standards of the D.E.K. and the 1940 standards of the Society were as follows—

Standard	1939 German	1940 British
1	0·8% Brilliant Wool Blue FFR extra	0·8% Brilliant Wool Blue FFR extra
2	1·0% Brilliant Wool Blue FFB extra	1·0% Brilliant Wool Blue FFB extra
3	1·2% Brilliant Indocyanine 6B	1·2% Brilliant Indocyanine 6B
4	1·2% Supramine Blue EG	1·1% Polar Blue G conc.
5	1·0% Cyananthrol RX	0·8% Solway Blue RS (= Cyananthrol RX)
6	3·0% Alizarine Light Blue 4GL	2·5% Alizarine Light Blue 4GL
7	3·0% Indigosol 06B	2·5% Soledon Blue 4BC powder
8	3·0% Indigosol Blue AGG	3·0% Indigosol Blue AGG

The British Committee is at present engaged on further minor revisions of the light standards, in particular standards 6 and 7.

(*Received 20th March 1951*)

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PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—II

The Design of Fading Lamps

J. Boulton and J. C. Guthrie

Some features of the past designs of fading lamps have been unsatisfactory, and a really efficient instrument probably does not exist, though some are better than others, largely on account of convenience of operation [1–6]. It may, therefore, be of interest to put down some basic considerations.

(i) With any kind of known equipment it is not possible to simulate sunlight fading with any degree of precision. In this country it is common practice to check fading lamp findings by exposures to sunlight (see e.g. Burgess [7]). The essential use of an instrument is for rapid comparative tests.

(ii) The practice of awarding "fading lamp hours", e.g. by docketing samples with the number of hours they resist exposure to an artificial light source, is to be strongly deprecated. It connotes equivalent behaviour in, and control of, instruments of the same make at different times in different places, and these conditions do not exist.

(iii) In the present state of our knowledge and of our accepted best practices of assessment, any fading machine should be put out simply and solely as a means of comparing a given pattern with an accepted standard pattern, and results must be stated in terms of the behaviour of the standard at the time of exposure of the patterns.

(iv) Nor is it possible to "standardise" exposures by the use of a single dyed standard whose exposure at any times to any lamp would give a factor for fading activity which could be applied to an "observed" fading time to give a "corrected" fading time. The behaviour of a coloured test paper, say, cannot be related at one and the same time to that of variously dyed wool, cellulose, nylon, leather, and so on. The existing B.S.I. standard patterns [8] are, it is true, prepared on a single substrate (wool). But they are not used in that way. They provide an agreed arbitrary *scale* for comparative performance of dyed patterns, and must not be used to "standardise" lamps by a time–fading activity relationship.

(v) With these conditions for the use of fading machines in mind, we consider that the outstanding features requiring attention in designing a lamp are—

(1) Temperature control
(2) Humidity control
(3) Relative movement of light source and exposed specimens
(4) Nature of light source
(5) Measurement of energy emitted by the light source.

The following detailed comments are based upon experience with the most modern (May 1949) American and one existing British instrument.

1. Temperature Control

The American instrument is capable of giving air temperatures in the exposure chamber within ± 2°F. of the working temperature chosen. The air temperature used is usually 100°F., but this is *not* the temperature of the surface of the sample. Careful measurements with a series of thermocouples showed that surface temperatures varied from 147 to 178°F., depending on both the colour of the fabric and also the type of dye used. This is borne out by a paper by Rees and Ogden [9] and applies to all fading lamps as well as sunlight.

It therefore appears improbable that sample temperatures in such a fading lamp will all be the same, and the criterion will probably have to be an air temperature constant within limits, say, of ± 2°F.

2. Humidity Control

Relative humidity control in the American instrument is not satisfactory, and it is possible that the American Association of Textile Chemists and Colorists stipulate a relative humidity not greater than 50% because the instrument cannot achieve this figure. Measurements of the moisture regains (percentages of moisture referred to the dry weight of the sample) of wool samples showed that values fell from approx. 16% to below 6% within a minute, and stayed in the region of 4–5% for the rest of the exposure.

From moisture absorption isotherms 4–5% moisture regain on wool is equivalent to a relative humidity of 10–15%. These low figures are in part due to the high surface temperatures mentioned under (1) above. The sample surface temperatures in sunlight fading can reach 160°F.; so the high surface temperature is not in itself an objection, and a water screen for cooling is not necessarily essential in a fading lamp. The amount of moisture to be added to produce higher humidities without a water screen is large, but this can be provided by means of steam or an atomiser, either of which could be controlled by a humidity-sensitive device.

Provision should also be made for "wet fading", which is frequently required, by keeping the samples saturated with water throughout the exposure; a spray arrangement has proved successful when applied to the two instruments already mentioned. Marney [10] has described a modification to the Calico Printers Association Ltd. fading lamp, which we have found to be very satisfactory.

The importance of relative humidity is once again stressed, and in fading lamps without control of humidity a most important factor is neglected. Some examples of the effect of humidity on fading are given in Cunliffe's original reports [8,11,12], and the matter is also referred to by Morton [13] and Lead [1].

3. RELATIVE MOVEMENT OF LIGHT SOURCE AND SPECIMENS

Owing to directional variations in intensity of the light from a carbon arc or other light source, rotation of either the source or the samples is essential. Experiments with commercial sources and a light integrator have shown that as much as twice the radiant energy can fall on one pattern as on another equidistant from the source but in a different radial direction. Given sound design, it would not appear to matter whether the lamp or specimen-holder is rotated, but keeping the latter fixed may be advisable from the point of view of the siting of controls, etc. Temperature and humidity controls should be operated from positions close to the sample surface, and may require to be carried on the sample holder. Further, if a recording light integrator is to be incorporated (see (5) below), the photocell (or thermopile or bolometer) must be close to the sample surface. With these in position, i.e. temperature- and humidity-sensitive controls and the photocell, designing an arrangement for rotation of the samples would be difficult. Alternatively, if the light source rotated about its axis, the only movable contact would be on the electrical supply to the arc. Any fluctuation in light intensity due to this latter sliding contact would affect the samples, and would also be recorded on the light integrator. On the other hand, with a fixed source and rotating samples, etc. the effect of any variation due to the sliding contact (on the photocell this time) would give rise to errors in the light energy recorded.

It is therefore considered that the samples, their controls, and the light integrator should be fixed and the light source made to rotate. It would not matter greatly whether all the samples were in one enclosure or whether several smaller compartments were used, each with its own temperature and humidity control. The former arrangement would be preferable for routine testing, where large numbers of samples are to be exposed under a given set of conditions.

4. LIGHT SOURCES

Consideration should be given to the gas arc [14,15] developed by Messrs. Siemens Electric Lamps & Supplies Ltd., which is a quartz discharge tube filled with xenon, argon, or krypton. The original is a 5-kw. water-cooled model, but a 3-kw. air-cooled type may be preferable for fading purposes. It is claimed that the gas arc gives a continuous spectrum which, in quality, approaches that of bright daylight, and it also runs at a reasonably low temperature.

Another possibility is the "I.G. accelerated light fading equipment" [16], where the light source consists of banks of tungsten filaments in nitrogen-filled tubes. Details are meagre in the reference quoted, but the possibility of using tungsten lamps for fading should not be overlooked. By the use of appropriate filters a fairly close approximation to daylight may be obtained, and this has been appreciated by the Commission Internationale de l'Éclairage (1931) in their choice of standard illuminants B and C for use in colour measurements [17].

Sadov and Vatulya [6] in their description of Irradiator SV claim to be able to carry out a fading and photodegradation test in 20 hr. The light source consists of banks of mercury-in-quartz lamps type PRK–2, which they claim to imitate natural daylight. On the other hand, Rabe [4] states that entirely unsatisfactory results are given with quartz mercury lamps, since their spectral distribution curves differ essentially from those of daylight.

The enclosed-type carbon arc is used on most British and American fading lamps at present. The relative merits of carbon and mercury arcs as light sources for fading have been discussed by Cooper and Hawkins [18].

5. RECORDING LIGHT INTEGRATORS

It is not suggested that a light integrator is essential, but it has been found to be definitely useful in keeping check on the variable output of existing lamps, and has enabled similar exposures to be given to similar samples at different times. It is of great value where an instrument is in use for research purposes rather than for purely routine comparative testing.

Several light integrators have been described (e.g. Davenport [19], Atherton [20]). An instrument developed in this laboratory will be described elsewhere, and Vickerstaff and Tough [21] quote seven other references. If the samples to be exposed and their temperature and humidity controls are stationary, all that is required on the fading lamp is a small window for insertion of the photocell.

Should it be considered that sufficiently close control of temperature and relative humidity is attainable with samples rotating round a stationary light source, a light-weight light integrator would require to be rotating with the samples.

In either event, the light integrator may be used to control exposure times by operating a relay, which would switch off the power to the light source, after a certain amount of light energy had been recorded.

The spectral distribution of daylight and artificial light may vary, and a light integrator employing a single photocell gives a value of the total light energy with no information as to its distribution according to wavelength. This also applies to the thermopile and bolometer. If information is required concerning the amounts of light energy falling on the samples in different wavelength bands, a series of photocells, filters, and associated circuits will be necessary.

To sum up, in a fading lamp the temperature and humidity controls should be capable of providing a variety of conditions with as wide limits as possible and with provision for wet fading. These are essential, particularly if the lamp is to be used for research purposes. Possible tolerances would be $\pm 2°$F. (air) and $\pm 4\%$ R.H. Routine tests would normally be carried out under a fixed set of conditions, but this should not imply that the lamp would operate satisfactorily only under these conditions. Temperature and relative humidity controls as well as the samples should preferably be stationary on the circumference of a circle with the light source rotating about its axis at the centre. The carbon arc (2000 kw.) is probably the best light source available at present, though consideration should be given to the possible use of the gas arc or tungsten filament lamps. The use of a light integrator for recording light energy incident on the samples, which may be attached to any fading lamp, is strongly recommended.

* * *

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MANCHESTER

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Felting of Permanganate-treated Wool

The Editor

SIR,

In a recent paper Dr. P. Alexander[1] discusses at some length the peculiar behaviour of permanganate-treated wool in milling and felting. The explanation suggested is that "the softened scale substance acts as an adhesive and sticks to the next scale, thereby masking the tips". This adhesion is sometimes broken, and the tips become free. Whether it is broken or not will determine the milling and felting behaviour of the treated wool. Dr. Alexander then continues to give some frictional measurements to support his idea. The fibres treated in this way did not give any differential frictional effect (D.F.E.) measured by the "violin bow" method[2], while in the Lipson capstan method[3] much of the D.F.E. was retained. "In the former method the fibres are kept straight and no opening of the scales occurs, but in the other method the fibres are bent; *this breaks the seal, frees the scale tip, and restores the* D.F.E. (our italics).

Dr. Alexander points out that this "hypothesis is purely tentative", but even so two remarks about his conclusions ought to be made—

(1) No dimension for the cylinder used in the Lipson method is given, but a reasonable value would be 5 mm. (Lipson used 8 mm.)[3]. Assuming the fibre diameter to be 20μ. and the length of a scale 25μ, simple calculation shows that the deviation of a scale tip, in relation to the tips of adjacent scales when the fibre is bent over the cylinder, will be only 0·1μ. This value is of the same order as the height of the scale tip and is decidedly too small to "break the seal". Dr. Alexander mentions Mr. Millson, of the American Cyanamid Co., who has shown that in extreme cases the scales break off. Mr. Millson, however, is working with a radius of curvature of the fibre axis which is about 100 times less than that obtained in the ordinary Lipson method[4].

(2) Even if such a small deviation outwards as calculated above would break the scales free, one is bound to use the concave side of the fibre, where the deviation is inward. This inward motion of the scale tip should not cause the scales to break off, but instead should further accentuate the adhesion of the tip.

Yours faithfully

JOEL LINDBERG

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GOTHENBURG
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26th January 1951

The Editor

SIR,

Before dealing with the points raised by Dr. J. Lindberg, I would like to summarise the experimental findings which require an interpretation.

When wool is treated with acid solutions of permanganate, as well as a number of other oxidising agents, including sodium hypochlorite in the pH range 8·5–9·5, it acquires resistance to felting when exposed to a mechanical treatment in neutral or alkaline solution such as occurs in a household washing machine, which does not involve a high degree of compression. The same material when squeezed hard as in milling by hand, or when pounded by hammers in a milling stock, felts as much as it did before treatment. It is evident, therefore, that there exist at least two distinct types of mechanical action which can bring about felting, and which for simplicity I have referred to as "washing" and "milling" respectively. In the former the fibre migration necessary for felting is produced by the flow of water, whereas in milling the alternate compression and relaxation bring about the movement.

It is generally accepted that scaliness or D.F.E. of wool fibres is necessary for the relative movement of fibres to bring about felting. It was shown that the D.F.E. of permanganate-treated fibres, when measured by the violin bow method, which involves no distortion of the fibres, was greatly reduced; hence wool so treated would not be expected to felt, and its behaviour during milling was anomalous. However, the D.F.E. of wool fibres when determined by the Lipson method, which involves sharp bending, was not greatly changed on treatment with permanganate. It is very probable that the two sets of observations are related, and that after reaction with permanganate the fibres do not felt and have no D.F.E., but on distortion the tendency to felt, and the D.F.E. are restored.

The photomicrographs of Millson and Turl[4] showed that on bending a fibre the scale tips protrude, and this suggested the theory that acid permanganate softens the scales and makes them adhere to one another. On distorting the fibre, however, the tendency of the scale tips to stick out results in a breakdown of the seal and a reappearance of the scale tips, which are necessary for the D.F.E. and for felting.

Dr. Lindberg does not believe that the distortion of a wool fibre during a measurement by the Lipson method is sufficient to free the scale tips. However, he considers that the bending movement is exercised over one scale only, whereas a number of scales are probably all stuck together, so that the deviation may be much larger than Dr. Lindberg calculates. Moreover, inspection of the photomicrographs of Millson and Turl[4] shows that, even at quite small radii of curvature such as exist some distance away from the peak of the bend, the scale tips protrude. It is therefore not at all improbable that during the measurement of the D.F.E. by the Lipson technique the fibre is sufficiently distorted for the scale tips to break the seal which holds them down and prevents them from producing a D.F.E.

The second point of Dr. Lindberg, that a seal would not be broken on compression, is incorrect, since a weak join is broken by any distortion, be it compression or extension. Direct proof, however, is again provided by Millson and Turl[4], who show that dyeing occurs preferentially at points where the scale structure is opened up. On bending a fibre, photomicrographs show preferential staining on both sides of the loop. It is thus clear that on the concave side, i.e. under compression, the scale tips are forced out, and hence should be capable of breaking the postulated seal.

I have not changed the view that my theory of the physical properties of wool fibres oxidised with permanganate is purely tentative, and should certainly be put to direct experimental test, probably by an electron-microscopic investigation. Purely theoretical arguments which are not supported by the few established facts at present available are not, however, likely to contribute to and understanding of the problem.

Yours faithfully

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Notes

Proceedings of the Council

At a meeting of the Council, held at the offices of the Society, 32–34 Piccadilly, Bradford, on 14th February 1951, the proceedings included the following items of interest—

CHEMICAL SOCIETY JOINT LIBRARY COMMITTEE— It was resolved that Dr. T. H. Morton be invited to represent the Society on this body.

HIGHER TECHNOLOGICAL EDUCATION— It was resolved to accept the recommendation of the Diploma Committee, that the Minister of Education be informed that the Society approved the general principles on which the report on the *Future Development of Higher Technological Education* was based, and would be glad to co-operate with the proposed Royal College of Technologists in the furtherance of technological education.

ANNUAL REPORT AND BALANCE SHEET— Drafts were approved for submission to the Annual General Meeting 1951.

XIITH INTERNATIONAL CONGRESS OF PURE AND APPLIED CHEMISTRY— It was reported that the Society had received an invitation to be represented at this Congress, to be held in New York on 10–13th September 1951.

MEMBERSHIP— Twenty-seven applications for ordinary membership, ten for junior membership, and one for readmission were approved.

RETIRING MEMBERS OF COUNCIL— The President expressed his thanks to retiring Vice-presidents

and Ordinary Members of Council, and expressed the hope that they would maintain their active interest in the Society.

Meetings of Council and Committees
April

Council— 11th

Publications— 17th

Colour Index Editorial Panel— 2nd

Fastness Tests Co-ordinating— 10th

Dyeing Affinity of Regenerated Cellulose— 17th

Deaths of Members

We regret to report the loss by death of Mr. R. L. H. Carlile, Mr. R. E. Hale, and Mr. J. A. Stott.

The Society of Dyers and Colourists' Fastness Tests Investigations

The Fastness Tests Co-ordinating Committee intends to publish papers from time to time dealing with its current or past investigations on fastness tests and standards. Some of these will describe the work of individuals, while others will be due to the work of the Committee as a whole or of one of its technical subcommittees.

The first two papers under this scheme relate to light fastness. The first (p. 188) contains a short documented history of the development of the present S.D.C. light standards from the inception of the original fastness scheme in 1927 to the present day. The object of this is to record facts which are tending to be forgotten, particularly overseas, with the passage of years. For this reason the Committee is hoping to effect simultaneous publication in France, Germany, Switzerland, and the U.S.A.

The second paper (p. 190) expresses the views of the members of the Light Fastness Subcommittee on the design and use of fading lamps.

It is hoped to publish further papers of this type in the near future.

Tentative Definitions

The Terms and Definitions Committee of the Society will welcome comments on the following tentative definitions—

1. AFFINITY

AFFINITY is the attraction between a fibre or other substrate and a dye or other substance under the precise conditions of test whereby the dye or other substance is selectively extracted from the application medium by the fibre or other substrate.

It should be noted that in physical chemistry the term "affinity" is equivalent to "free energy", the standard affinity of a reaction being given by the equation—

$$-\Delta G^\circ = RT \ln K$$

where R is the gas constant, T the absolute temperature, and K the equilibrium constant of the reaction.

2. COLOUR

(*a*) That characteristic of the visual sensation which enables the eye to distinguish differences in its quality, such as may be caused by differences in the spectral distribution of the light rather than by differences in spatial distribution or fluctuations with time.

(*b*) As (*a*), but applied directly to the stimulus or the source (primary or secondary) giving rise to the sensation. For brevity the stimulus is often referred to as the colour.

(*c*) That property of an object or stimulus, or quality of a visual sensation, distinguished by its appearance of redness, greenness, etc., in contradistinction to whiteness, greyness, or blackness (i.e. chromatic colour in contradistinction to achromatic colour).

The above definitions of colour are those recommended by the Physical Society Colour Group (*Report on Colour Terminology*, p. 4). It should be noted, however, that in the dyeing industry greys but not blacks are regarded as colours.

South African Post for Mr. R. C. Palmer

Mr. R. C. Palmer, M.A., has been appointed Director of Research to the new South African Wool Textile Research Institute, which is intended to serve the Union wool manufacturing industry and act as a link with South African pastoral research workers. Since 1937 he has been with the Wool Industries Research Association as head of the research section dealing with wool washing, carding, and combing.

Before going to Torridon he did research work on surface chemistry problems with Professor Eric Rideal at Cambridge.

New Appointment of Mr. P. A. Wells

Mr. P. A. Wells, M.A., M.Sc., has been appointed Deputy Secretary and Secretary-elect of the Royal Sanitary Institute. He was General Secretary of the Society from 1945 to 1947, and then became Director of Education to the International Wool Secretariat.

British Plastics Convention
London, 6–16th June 1951

The first trade exhibition and convention to represent Britain's entire plastics industry will be held in the National Hall, Olympia, London, during June. Among papers of special interest to members of the Society are—

Friday, June 8th, 2-30–4-0 p.m.

Textile Applications of Aminoplastics — A. R. Smith

Plastics and their Application to Equipment in the Textile Industry — A. Beecham

Monday, June 11th, 10-15 a.m.–12-30 p.m.

The Physicochemical Background of Synthetic Fibres — Professor E. K. Rideal

Terylene — A. Renfrew

Forms of application for admission tickets and further particulars are obtainable from the Exhibition Manager, *British Plastics*, Dorset House, Stamford Street, London S.E.1.

OBITUARY NOTICE

Thomas Saville Whittle

His wide circle of friends will mourn the death of Thomas Saville Whittle, which occurred on 30th December 1950 at his home in Swinton, Manchester. He had been associated with dyes and chemicals during the whole of his lifetime, and was a member of the Society. Born in Chorley in 1881, he started work at the Low Mill Print Works, Whittle-le-Woods, and in his spare time studied at the Salford Royal Technical College.

Anxious to improve his position and experience, he decided to go abroad, and spent the years 1900–1908 in the Colouristic Department and Dyehouse of Leopold Cassella & Co. at Mainkur, Germany. Here he became acquainted with the dye industry and the wide application of dyes to textiles generally. When he returned to England in 1908, he continued his association with Cassella's through the firm of Brown & Forth Ltd., who represented them.

In 1915 he founded his own firm of T. Saville Whittle, later acting as the sole importer of Cassella products in Lancashire. This continued until the formation of the I.G. Farbenindustrie A.-G. During World War I he joined the Army, serving with the Honourable Artillery Company, and later he carried out extensive work at the University of London on poison gases. In 1923 he formed the firm of T. Saville Whittle (Export) Ltd., and developed a valuable connection with textile mills in Brazil.

Mr. Whittle had, apart from his business interests, many other activities. He was for many years a Councillor for the County of Lancaster, he was Justice of the Peace and for a long period Chairman of the Visiting Committee at Strangeways Prison. He took an active interest in sport, was President of the Swinton Rugby League Football Club and of the Lancashire and Cheshire Cricket League, as well as a Director of the Haydock Park Racecourse Co. Ltd.

He is survived by his widow and his only son, Mr. Thomas Whittle, who is a director of both firms.

A. F. KERTESS

New Books and Publications

The Manufacture of Intermediates and Dyes

An Introduction to Works Practice

By G. H. Frank. Pp. viii + 177. London: Constable & Co. Ltd. 1950. Price, 15s. 0d.

This book is a contribution to a subject the existing literature of which, in England, is, as the author states, scanty. He ought, however, to have included in his introductory review P. W. Vittum's translation of H. E. Fierz-David and L. Blangey's *Fundamental Processes of Dye Chemistry*, published as recently as 1949.

Mr. Frank divides his book into five parts— I *General Notes*, which deals all too briefly with services, plant, materials of construction, and some of the hazards of the industry. II *Benzene Series*, which contains detailed accounts of the preparation of benzene, toluene, and xylene from coal tar and the manufacture of many derivatives, including chlorobenzene, nitrobenzene, aniline, and benzidine. The manufacture of three sulphur dyes— Black, Brown 2G, and Blue— is also included in this section. III *Naphthalene Series*, which gives methods for the large-scale preparation of most of the important naphthols, naphthylamines, and their sulphonic acids. This part also includes a chapter on Azo Dyes, which contains brief descriptions of a typical azo unit, its mode of working, and methods for making Bismark Brown, Litho Red R, Era Chrome Brown MW, and Acid Blue 2B. IV *Anthracene Series* is much shorter than the previous two parts, the dozen or so anthraquinone derivatives, Alizarin Blue Black B, and some seven vat dyes, including Blue RS, Violet BN, and Dark Blue BO, being only briefly treated. V *Analysis* includes methods of estimating moisture, of testing benzene, toluene, nitrobenzene, various sulphonic acids, and Sulphur Blacks.

The works chemist concerned with the making of products included in the book will be interested to compare details given with his own experience. To the student the book will be useful, though here a more adequate treatment of the actual plant used would have been an advantage. For example, works methods of filtering, particularly those using the filter press (not "pressure filter", a term used in the book, which is usually reserved for something quite different), merit considerably more space in a book of this sort than the present author has given. Of all equipment used in the industry the filter press often proves to be the most puzzling for the student coming into the works for the first time. In the same section, too, noticeable omissions are the absence of any reference to the now widely used rubber diaphragm valves, and no mention of the hardwoods like Greenheart which are now finding increasing use in the industry. It is slightly disappointing in a book of so practical a nature to find once again in print an unsatisfactory working method like the one given for the separation of unchanged anthraquinone in the preparation of anthraquinone-α-sulphonic acid.

Though there is lack of balance between its various parts, the book as a whole is well arranged, easy to read, adequately indexed, well printed, and illustrated with sixteen diagrams and three plates.

R. K. FOURNESS

Scientific Russian

A Text-book for Classes and Self-study

By James W. Perry. Pp. xxix + 816. New York and London: Interscience Publishers 1950. Price, $7.50 or 60s. 0d.

In spite of the importance attained by Soviet scientific work, the British scientist who can read

Russian remains a rarity. Many, no doubt, shrink before the prospect of learning what they regard as an obscure tongue of highly exotic character clothed in repellent hieroglyphs. Let them be assured, however, that under Professor Perry's guidance they will find the road much easier than they anticipated. The author, now Associate Professor of Modern Languages at the Massachusetts Institute of Technology, is also a chemist and is co-author of a recent book on surface-active agents. He has combined his fields of activity in admirable fashion in the very lucid reading passages contained in the book under review. They range over many fields of interest to the physicist and chemist, though the biologist receives scant attention.

The early lessons on nouns and adjectives are devoted to international scientific terms, and through these not only the alphabet, but also many formative suffixes, should be rapidly and painlessly assimilated. Russian roots are introduced gradually. Later, verbs are treated in similar fashion, and the grammar is developed on more or less traditional lines, word-forms and constructions not normally met in scientific writings being excluded. Additional grammatical information may be gleaned by studying the numerous sentences with interlinear translation, but there are a number of points that should have received specific mention— e.g. the use of negative pronouns with prepositions; the use of verbal nouns consisting of a verb stem with or without the ending *-ka*, a type very common in technical literature (cf. *otbelka*, "a bleach", with *belenie*, "bleaching"); the use of adjectives that form an extension to the category discussed in §278, but are derived from perfective verbs (in sentences on pp. 456–457, which are rather artificial and would scarcely be used in practice, such an adjective, e.g. *primenimy*, could with advantage replace a group of three words). Each lesson ends with a series of English sentences for translation; some instruction in written script would have been useful here.

The text is accented throughout (there is no golden rule for the position of the stress in Russian). This is a good feature, for, although the primary aim is a reading knowledge of the language, the student is rightly advised to associate a sound with each word, which should be read without conscious translation. Also, it is possible that he may require an understanding of the spoken language. For these reasons some guide to the pronunciation of chemical symbols and of such intractable expressions as $1{\cdot}23 \times 10^{-5}$ might well have been included. Errors in accentuation are rather frequent.

Few errors detracting from the general usefulness of the work were detected. Occasionally imperfective aspect is used when perfective would be more appropriate, e.g. p. 411, lines 10 and 11; also p. 257 (*sozdavat'*). In §170 α-rays are regarded as electrons and β-rays as helium nuclei, but in §182 the opposite view is held.

This is the first adequate treatment of scientific Russian for English-speaking students. It is an excellent production and may be confidently recommended as a highly satisfactory treatment of the subject.

A. E. STUBBS

The Setts of the Scottish Tartans

with descriptive and historical notes

By Donald C. Stewart. Pp. xvi + 125. Edinburgh: Oliver & Boyd 1950. Price, 70s. 0d.

Everyone connected with the textile industry has noted with interest the remarkable increase in popularity of plaids and tartans during these past few years. There is still no sign of this interest diminishing. Woven in wool in the traditional style or used as the basis of freely adapted checks in silk or rayon, the original family or clan tartan has shown, in its colour and decorative possibilities, a useful and lasting type of pattern for dress-materials in the home market and especially for overseas buyers.

The weaving of the typically Scottish tartan has existed as both a craft and an industry certainly since the early eighteenth century and, although its prime purpose was the clothing of the clansmen and the Highland regiments, it was regarded in England as good material for bed-hangings, coverings, etc. Since the middle of the nineteenth century the wearing of tartan in Scotland by both men and women has gradually increased, and the result has been a constant demand for ancient and authentic clan patterns. Despite the undoubted antiquity of tartan, it is unfortunately true that it is difficult and, at times, impossible to determine with any kind of accuracy the setts of the various families and clans. Many books have been published on this subject (an exhaustive list is given by Mr. Stewart), but the confusion or uncertainty has remained.

It can be stated that the book under review is probably the most authoritative volume yet published. Full research has been made, with sifting of all available sources, literature, old portraits of the clan chiefs and Scottish nobility, and existing examples of ancient tartan materials; the author has also woven examples himself from traditional recorded setts, and formed opinions regarding the authenticity of patterns from the accumulated evidence. The setts are illustrated by coloured strips— reproduced by lithography— showing the relative width of the coloured stripes in the cloth and complete counts of threads for each pattern illustrated are given in the text. This method is an adaptation of the old system of the tartan weavers of winding the requisite numbers of coloured threads on a narrow stick. In addition, eight plates in photolithographic process show facsimiles of tartans woven by the author from old examples. The illustrations are uniformly excellent in correct colour renderings and, combined with the full notes of the text, provide a complete and comprehensive study of present-day knowledge of the subject of tartan design.

It is an expensive book but it is a fine example of modern book-production. To the weaver and manufacturer or to the person interested historically or otherwise in the general subject of

tartans and plaids it should provide a first-class book of reference. It should inspire also those weavers who are interested not only in the reproduction of the ancient setts but in the evolution of new designs based on traditional forms.

J. CHIRNSIDE

Abstracts from British and Foreign Journals and Patents

(The Titles of Patents are abridged and modified)

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Keeping Nozzles from being choked by Dust or Fibre. W. W. Spooner. *B.P.* 649,256.

Continuous Processing of Filaments. I.C.I. Ltd. and R. Lipscomb. *B.P.* 649,997.

Continuous lengths of artificial filaments, e.g. nylon or polymethylene terephthalate, are laced in a zig-zag path between two lines of pegs spaced apart and projecting from one or two endless conveyors which carry the pegs and filament through various treating zones, if desired diverging at some point to draw out the filament.

J. W. B.

Treating Loose Fibres with Liquids. Johnson & Johnson (Gt. Britain) Ltd. *B.P.* 649,451.

A stream of the liquor impinges upon successive portions taken from the mass of fibres so as to wet the individual fibres. The wet fibres are then reassembled into a mass, in which they are retained until the process is completed. The various portions of the fibre mass and the treating liquor move in the same direction, but movement of the former is repeatedly retarded while the liquor flows through them to agitate the fibres. Suitable apparatus is described. The procedure is suitable for boiling or scouring cotton to render it absorbent. C. O. C.

Treating Hanks with Liquids. A. Ashworth. *B.P.* 649,665.

The hanks are hung in a chamber and packed so tightly that they form a floor dividing the chamber into two parts. Liquid is supplied to the top of the chamber, where it maintains a head over the hanks, through which it percolates slowly. This gives very even treatment of the hanks with economy in the volume of water used.

C. O. C.

Continuous Wet Treatment of Open-width Fabric. E. F. Williams and W. J. Brayden. *B.P.* 649,771.

The fabric is passed under longitudinal tension past a zone in which a pool of liquor is maintained in direct contact with one face of the fabric. A uniform layer of the liquid is continuously withdrawn from the pool on to a narrow area extending across the fabric. This layer of liquid is drawn through the fabric by means of a high vacuum acting on the other face of the fabric and solely against the atmosphere. Suitable apparatus is described.

C. O. C.

Treatment of Paper, Cloth, etc. W. W. Spooner. *B.P.* 649,308.

In treating cloth, etc. in apparatus of the type where the cloth is displaced continuously or intermittently in a vertical plane or planes and is at the same time treated by streams of liquids or gases, e.g. from nozzles placed on one or both sides of the material, the material is borne along by guides carried by spaced parallel levers pivoted in alternate disposition on opposite walls of the treatment chamber, these levers being interconnected by links.

C. O. C.

Coating and Laminating Cloth, Paper, etc. E. J. Carter and W. M. Scholl. *U.S.P.* 2,525,863.

Drying. Sutcliffe Speakman & Co. Ltd. and W. E. Edwards. *B.P.* 649,527.

The drying air is passed through or into contact with the material to be dried, e.g. clothes in a rotary drier, and then, when partly saturated in one operation by treating one batch, is used, under conditions to prevent deposition of absorbed liquid, either to retreat the same batch of material or to treat a second wholly wet batch. C. O. C.

Drying Printed, Painted, etc. Sheet Material. Timsons Ltd. and E. A. Timson. *B.P.* 649,606.

One or more flat hotplates are arranged in the path of the material to be dried and can be moved to and from a drying position close to or touching the material.

C. O. C.

Cloth Rigging, Measuring, and Plaiting. Sellers & Co. (Huddersfield) Ltd. and E. R. Sellers. *B.P.* 649,669.

Coating Webs. J. Waldron Corpn., C. A. Dickhaut, and B. R. Newcomb. (X, p. 204.)

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

PATENTS

Starch Pastes. Girdler Corpn. *B.P.* 649,324.

An aqueous starch slurry is continuously fed under positive pressure endwise through an elongated heating zone. Here it is agitated and indirectly and rapidly heated so that it gelatinises. The resultant paste is continuously discharged from the heating zone into a cooling zone, where it is rapidly cooled at such a rate as to maintain the state of gelation obtained in the heating zone. This ensures uniformity of product and enables precise yet flexible control of the type of paste produced. C. O. C.

Antistatic Treatment for Synthetic Linear Polymer Yarns. British Nylon Spinners Ltd. and G. H. L. Andrew. *B.P.* 649,877.

Application of a solution of sodium chloride in a hygroscopic liquid aliphatic polyhydroxy compound of b.p. > 200°c., e.g. 2% (wt./vol.) NaCl in diethylene glycol containing 20% water by vol. C. O. C.

Acetone Resins for Laminating Paper, Cloth, etc. Bakelite Corpn. *B.P.* 649,304.

Heat-hardenable resins are obtained by condensing 1–2 mol. of formaldehyde with 1 mol. of acetone in presence of enough of a secondary amine soluble in the reaction mass to maintain the latter at pH 9·5–11·5 during the greater part of the condensation. C. O. C.

Polyvinyl Acetate Adhesive Compositions. Shawinigan Chemicals Ltd., G. O. Morrison, T. P. G. Shaw, J. D. P.-E. Mercier, and H. M. Collins. *B.P.* 649,505.

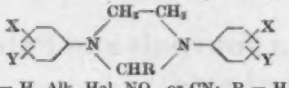
A tacky, non-slip, water-resistant adhesive comprises a stable aqueous emulsion of polyvinyl acetate, which is formed by emulsion polymerisation and contains as emulsifiers 0·25–1·0% gum tragacanth, 0·25–0·75% partly hydrolysed polyvinyl acetate, and an anionic surface-active agent, e.g. 0·1% sodium dioctyl sulphosuccinate, a plasticiser effective in amount ranging from 20 to 180% by wt. of the polyvinyl acetate, e.g. dibutyl phthalate, and a substantially water-immiscible solvent. J. W. B.

Fatty Acid–Aliphatic Amine–Polyglycol Combinations useful as Finishing and Softening Agents. Arkansas Co. Inc., A. A. Cook, and I. Wolf. *U.S.P.* 2,525,771.

A fatty monocarboxylic acid of 12–24 C (1 mol.) is heated with a water-soluble primary or secondary aliphatic amine (1–1·5 mol.) containing at least 2 amino groups or 1 amino group and 1 or more OH groups at 165–205°c. for 1–2 hr. The product is then heated with a polyethylene glycol of mol. wt. 200–1500 (0·1–1·0 mol. depending upon the mol. wt.) at 150–175°c. for 1 hr. The final product is highly complex and includes esters, amides, amino-esters, and usually small quantities of free polyethylene glycol. It is used as a finishing or softening agent for cotton and rayon using 2–10% on the weight of the fibre or 0·1–1·0% aq. soln. at pH 3–8 below 100°c. C. O. C.

1:3-Diphenyliminazolidine and Certain Derivatives thereof as Gas Fading Inhibitors. General Aniline & Film. Corpn., H. W. Grimmel, and A. Guenther. *U.S.P.* 2,525,898.

The colourless water-insoluble compounds of formula—



(X and Y = H, Alk, Hal, NO_2, or CN; R = H, Alk, Ar, or a furyl radical free from solubilising groups), e.g. 2-methyl-1:3-diphenyliminazolidine, are gas-fading inhibitors substantive to organic derivatives of cellulose. They are suitable for application in the dyebath. C. O. C.

Stripping Vat-dyed Textiles. Arkansas Co. Inc., A. A. Cook, and I. Sapers. *U.S.P.* 2,525,770.

The products obtained by heating not < 1 mol. of a non-hydroxylated polyamine containing 4–10 C and 3–6 N with a high-mol.wt. fatty acid of 12–32 C at 160–230°c. for 1–3 hr. and then cooling rapidly to 90–100°c. are excellent aids in stripping vat dyes, particularly those of the anthraquinone class. C. O. C.

Gelation of Drying Oils. L. Frenkel Ltd. and H. W. Chatfield. *B.P.* 648,934.

The addition of < 25% of an aluminium alkoxide of an alcohol containing > 3 carbon atoms, e.g. octanol, to animal and vegetable drying or non-drying oils promotes the rapid gelation of deposited films or coatings. E. C.

2-Nitroamino-Δ^2-1:3-diazacycloalkenes—Dye Intermediates, Dyeing Assistants, Vulcanisation Accelerators. Honorary Advisory Council for Scientific and Industrial Research (Canada), A. F. McKay, and G. F. Wright. (IV, p. 200.)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Study of the Sulphonation Reaction. XV—Certain Properties of Naphthalenemonosulphonic Acids. A. A. Spryskov. *J. Gen. Chem.* (*U.S.S.R.*), **20**, 2101–2104 (Nov. 1950).

The water of crystallisation associated with α- and β-naphthalenesulphonic acids after drying over various agents is determined. The anhydrous α-acid, m.p. 139–140°c., was prepared for the first time. Data are given relating to the solubilities of the free acids and their Na salts in water and in aqueous solutions of acids and salts. A. E. S.

Monoazo Dyes from *m*-Nitrobenzoic Acid. I—Preparation of *m*-Nitrobenzoic Acid. T. Nishi and H. Toki. *J. Soc. Chem. Ind. Japan*, **45**, Suppl. binding, 37–38 (1942) [in German]: *Chem. Abs.*, **44**, 11,099 (25th Nov. 1950).

Methods are given for the simultaneous nitration and oxidation of toluene; e.g. at 15°c. 25 c.c. toluene was stirred over a period of 3 hr. into a mixture of 18 c.c. fuming nitric acid and 63 c.c. sulphuric acid. The stirred mixture was heated for 1 hr. at 40°c. and 1 hr. at 90°c. (temperatures > 100°c. led to explosions). The crude product (30 g.) was purified by dissolving in aq. caustic soda, treating at 50°c. with calcium hypochlorite, and reprecipitating with hydrochloric acid, to yield 18 g. purified *m*-nitrobenzoic acid. When the nitration temperature was kept at 40°c. for 8–12 hr. appreciable amounts of *m*-nitrobenzaldehyde were formed, whereas if the nitration mixture is finally heated for 10 hr. at 100°c., the principal product is 3:5-dinitrobenzoic acid.

II—Monoazo Dyes from Aminobenzoic Acids. T. Nishi and I. Nakajima. *Ibid.*, 38–39.

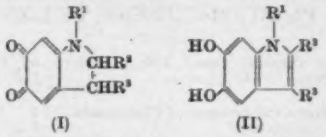
The three isomeric aminobenzoic acids were diazotised and coupled with J acid to give substantive orange–red dyes. The *ortho* derivative had the highest substantivity and the *meta* derivative the lowest. E. S.

Azo Dyes from 1:5-Naphthylenediamine. T. Nishi and H. Toki. *J. Soc. Chem. Ind. Japan*, **46**, Suppl. binding, 172 (1943) [in German]: *Chem. Abs.*, **44**, 11,100 (25th Nov. 1950).

Azo dyes from 1-5-naphthylenediamine and J acid stain cotton blue–violet in presence of Glauber's salt. Introduction of an aryl group into the NH_2 group of the J acid improves the affinity slightly, while introduction of an alkyl group improves it markedly. E. S.

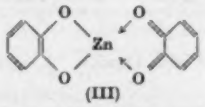
Synthesis of 5:6-Dihydroxyindole Derivatives—an Oxido-reduction Rearrangement catalysed by Zinc Ions. J. Harley-Mason and J. D. Bu'Lock. *Nature*, **166**, 1036–1037 (16th Dec. 1950).

Compounds of type I are rapidly converted to type II by the action of zinc acetate in aqueous solution, the rearrangement providing a convenient synthetic route to dihydroxyindoles which are of interest as melanin intermediates. In one example 3:4-dihydroxyphenylalanine is



(I) (II)

treated with 4 mol. equivalents of potassium ferricyanide and sodium bicarbonate to give a deep red solution of a quinone (I— $R^1 = R^3 = H$, $R^2 = COOH$), which, on treatment with excess zinc acetate, removal of precipitated zinc ferrocyanide by centrifuging, and ether extraction of the aqueous solution, gives an excellent yield of 5:6-dihydroxyindole (II— $R^1 = R^2 = R^3 = H$). The reaction is extended to halogen-substituted compounds; e.g. 2-iodoadrenochrome (I— $R^1 = CH_3$, $R^2 = I$, $R^3 = OH$) on treatment with zinc acetate and afterwards with sodium dithionite (hydrosulphite) gives 3:5:6-trihydroxy-1-methylindole. In support of the view that the effect is due to the intermediate formation and subsequent decomposition of a zinc complex, an insoluble dark green zinc complex (III) is obtained by the oxidation of catechol in presence of zinc salts.

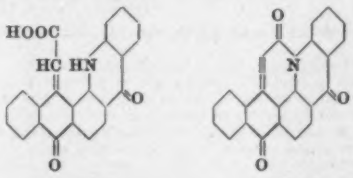


(III)

J. W. B.

Investigations in the Field of Polycyclic Compounds. XIII—Absorption Spectra of Anthrapyridone–Acridone, Anthraquinone–Acridone, Anthrapyridone, and some of their Derivatives. A. M. Lukin, P. M. Aronovich, and G. P. Brin. *J. Gen. Chem.* (*U.S.S.R.*), **20**, 2219–2221 (Dec. 1950).

Reference is made to a number of anthrapyridone–acridones, synthesised by two of the authors. These reacted with dil. NaOH with rupture of the pyridone ring. The constitution (I) assigned to the product of the reaction with alkali is now confirmed, in the case of the simple anthrapyridone–acridone (II), by comparing the absorption spectra of the following—II, the product of the reaction of II with alkali, anthraquinone–acridone, and various derivatives of II.



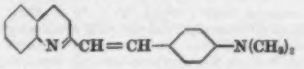
(I) (II)

A. E. S.

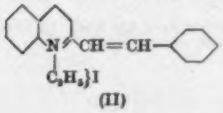
Molecular Interaction and Colour—Absorption Spectra of Molecular Compounds of Quinoline Salts with Aromatic Amines. V. A. Izmailsky and P. A. Solodkov. *Doklady Akad. Nauk S.S.S.R.*, **75**, 391–394 (21st Nov. 1950).

Examination of the absorption spectra of dyes containing separated chromophoric systems has shown that there is some degree of interaction between these systems. The authors have previously obtained small effects of a similar nature also for molecular complexes formed between pairs of dyes. They now show that under

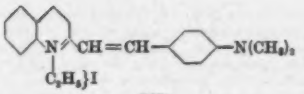
favourable conditions this latter effect may be very considerable, so that the molecular complex between A·[K]·H and B·[K]·H will give an absorption spectrum close to that of A·[K]·B ([K] represents a conjugated system; A and B are suitable end groups, e.g. $N(CH_3)_2$ or NO_2); i.e. the absorption max. of the complex will be at a longer wavelength and have a higher molar extinction coefficient than either of the separate components. In such a case the two π-electron systems are linked without an accompanying σ-linkage, and it is proposed to term such a linkage an *exo-bond*. Such a bond is formed e.g. between 2-*p*-dimethylaminostyrylquinoline (I) and 1-ethyl-2-styrylquinolinium iodide (II). This is shown by a comparison of the absorption spectrum of the complex with that of 2-*p*-dimethylaminostyryl-1-ethylquinolinium iodide (III).



(I)



(II)



(III)

The characteristics of the absorption max. for the above in dil. methanol soln. were found to be— $\lambda_I = 393$ mμ., $\varepsilon_I = 37{,}700$; $\lambda_{II} = 338$ mμ., $\varepsilon_{II} = 24{,}700$; $\lambda_{III} = 525$ mμ., $\varepsilon_{III} = 64{,}000$. When four parts of II and one part of I were added to methanol to give a 0·001 M. soln., the absorption max. was at 505 mμ. ($\varepsilon = 43{,}360$). Owing to the greater dissociation of the complex, λ and ε were less for an equimolecular mixture, being increased by an excess of either component; for the same reason the effect fell off rapidly with dilution. Effects of a similar nature were obtained also with mixtures of I with 1-ethylquinolinium iodide, with mixtures of dimethylaniline and 1-ethylquinolinium iodide, and with mixtures of dimethylaniline and II. The tendency for complex formation and bathochromic displacement is in the order—I > dimethylaniline, and II > 1-ethylquinolinium iodide. A. E. S.

Cyanine Dyes containing Fluorine. I— Cyanine Dyes from Derivatives of 6-Fluorobenzthiazole. A. I. Kiprianov and L. M. Yagupolsky. *J. Gen. Chem. (U.S.S.R.)*, **20**, 2111–2117 (Nov. 1950).

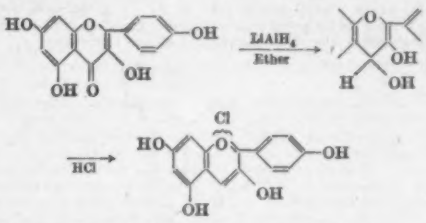
2-Methyl-6-fluorobenzthiazole and 2-methylthio-6-fluorobenzthiazole were synthesised and applied to the synthesis of 11 thiacyanine dyes, containing fluorine in the 6-position and an internuclear chain of 1–5 methin groups. The absorption spectra of these F-containing dyes were compared with those of the corresponding unsubstituted dyes, and in no case was there an appreciable difference in the positions of the absorption maxima. A. E. S.

Fluorescence of Magnesium Phthalocyanine and of Chlorophyll under Various Conditions— Structure of the Absorption and Fluorescence Spectra of Magnesium Porphyrin and of Chlorophyll. V. F. Gachkovsky. *Doklady Akad. Nauk S.S.S.R.*, **75**, 407–410 (21st Nov. 1950).

Experiments, similar to those reported for magnesium phthalocyanine (cf. J.S.D.C., **67**, 36 (Jan. 1951)), were carried out on an adsorbate of chlorophyll a. Under the conditions of degassing *in vacuo* partial decomposition occurred, leading to the formation of a magnesium porphyrin, which was studied also. The fluorescence spectra of the complexes formed by these two substances with oxygen, water, etc. are quite analogous to those found for magnesium phthalocyanine. In all these cases the absorption and fluorescent emission curves are related in mirror image fashion. From an examination of these curves the full scheme of electronic transitions is derived for each substance; in each case it involves two excited electronic levels. In addition to the spectra of magnesium porphyrin (which contains two double bonds outside the 18-membered conjugated ring) and of chlorophyll (in which one of these bonds is hydrogenated), the spectrum of bacteriochlorophyll (both bonds hydrogenated) was considered. Contrary to the view of Rabinowitch (*Rev. Modern Phys.*, **16**, No. 3–4 (1944)), hydrogenation is considered to lead not to the creation of new excited levels, but to the lowering of the lower excited level, with consequent shift of the absorption max. to longer waves. A. E. S.

Conversion of Flavanols into Anthocyanidins. R. Mirza and R. Robinson. *Nature*, **166**, 997 (9th Dec. 1950).

γ-Pyrones are reduced by lithium aluminium hydride to γ-pyranols, which on treatment with acids give pyrylium salts. Anthocyanidins are produced in this way from the appropriate flavanols, e.g. pelargonidin chloride from kaempferol—



The process works well with methoxylated flavones, though protection of OH groups is not essential; it also applies to the reduction of xanthones to xanthhydrols, and thence to xanthylium salts. Pinacol-like coupling of the molecules is avoided, and thus the product is uncontaminated by related pigments. J. W. B.

Methionine and the Formation of Pigment by Yeasts. N. S. Cutts and C. Rainbow. *Nature*, **166**, 1117 (30th Dec. 1950).

Formation of pink pigment by yeast 47 when growing in a defined salts–glucose–ammonia medium is associated with the presence of methionine and a relative deficiency of biotin. Omission of *p*-aminobenzoic acid from, or inclusion of adenine in, the growth medium also inhibits colour formation. Pink yeast crops are always connected with the presence of a diazotisable amine in the fermented medium. J. W. B.

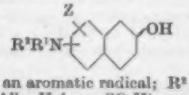
PATENTS

2-Hydroxy-1-naphthoic Acid. E. B. Higgins. *B.P.* 647,981.

β-Naphthol is stirred in tetrahydronaphthalene or diphenyl ether with metallic sodium at the m.p. of the latter, in an atmosphere of hydrogen or nitrogen, until a clear solution of naphthoxide is obtained. Carbon dioxide is next introduced into the refluxing mixture until the reaction is complete, and the resulting 2-hydroxy-1-naphthoic acid separated out of contact with air by filtering or distilling off the solvent. R. K. F.

β-Naphthol Derivatives as Colour Formers for Blue to Reddish-blue Phenazonium Dyes. General Aniline & Film Corpn., V. Tulagin, and W. A. Schmidt. *U.S.P.* 2,525,503.

Compounds of formula—

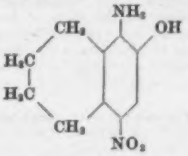


(R^1 = H, Alk, or an aromatic radical; R^2 = H or an acyl group; Z = H, Alk, Hal, or SO_3H) are used as colour formers for blue to bluish-red phenazonium dyes in conjunction with an *o*-phenylenediamine type of developer. C. O. C.

***ar*-1-Aminotetrahydro-2-naphthol Derivatives.** General Aniline & Film Corpn., C. G. Vogt, and J. M. Wilkinson, Jr. *B.P.* 647,209.

Derivatives of *ar*-1-aminotetrahydro-2-naphthol carrying NO_2, NH_2, Hal, OH, CN, CNS, or SO_3H in the 4-position

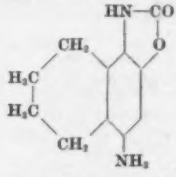
are prepared by hydrolysing the corresponding 4-substituted naphthoxazolone with aqueous alkali or mineral acid in an atmosphere of N_2 or CO_2. Thus *ar*-4-nitrotetrahydro-β-naphthoxazolone is refluxed in an aqueous suspension of lime for 20 hr. to give the compound—



R. K. F.

ar-Tetrahydro-β-naphthoxazolone Derivatives. General Aniline & Film Corpn., C. G. Vogt, and J. M. Wilkinson, Jr. *B.P.* 647,210.

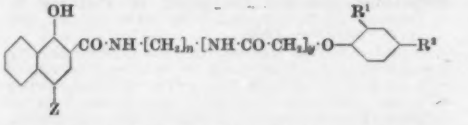
The β-naphthoxazolones used in the previous abstract are prepared by first nitrating *ar*-tetrahydro-β-naphthoxazolone in glacial acetic acid at 25°C. Reduction with hydrogen under pressure in ethanol in presence of platinum black gives the corresponding 4-amino derivative—



Other derivatives are made by diazotising and following with a Sandmeyer reaction. R. K. F.

Colour Couplers. Kodak Ltd., A. Weissberger, I. F. Salminen, and P. W. Vittum. *B.P.* 649,660.

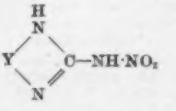
Compounds of formula—



(Z = H or Cl; R^1 = H or C_{1-4} Alk; R^2 = C_{1-4} Alk; n = 2–4; y = 0 or 1) used as colour formers with primary aromatic amino developers yield blue–greens of high purity and stability. C. O. C.

2-Nitroamino-Δ^3-1:3-diazacycloalkenes—Dye Intermediates, Dyeing Assistants, Vulcanisation Accelerators. Honorary Advisory Council for Scientific and Industrial Research (Canada), A. F. McKay, and G. F. Wright. *U.S.P.* 2,525,927.

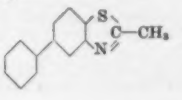
Compounds of formula—



(Y = a lower alkylene radical), e.g. 2-nitroamino-Δ^3-1:3 diazacyclopentene, are useful as dye intermediates, dyeing assistants, and vulcanisation accelerators. They may be prepared in good yield by treating a diacid salt of the appropriate alkylenediamine with an alkali-metal salt of nitroguanidine. C. O. C.

2-Methyl-5-phenylbenzthiazole Methin Dyes. Kodak Ltd., L. G. S. Brooker, and G. Van Zandt. *B.P.* 649,725.

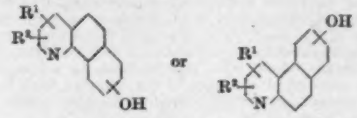
2-Methyl-5-phenylbenzthiazole—



can be made into quaternary salts from which methin dyes can be prepared. It is obtained by reducing bis-3-nitro-4-diphenylyl disulphide with Zn and acetic acid and then acetylating. C. O. C.

Benzoquinolines as Colour Formers for Phenazonium Dyes. General Aniline & Film Corpn., V. Tulagin, and W. A. Schmidt. *U.S.P.* 2,525,502.

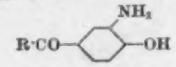
Compounds of formula—



(R^1 = an auxochromic group; R^2 = H or Alk; OH is in the β-position of the naphthalene ring system) yield blue-green phenazonium dyes when used as colour formers in conjunction with an *o*-phenylenediamine type of developer. C. O. C.

oo′-Dihydroxyazo Dyes containing a Keto Group. Ciba Ltd. *B.P.* 649,656.

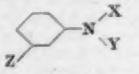
oo′-Dihydroxyazo dyes are made by diazotising a compound of formula—



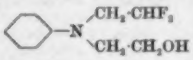
(R = Alk or Ar free from *o*-hydroxycarboxy), and coupling with suitable components. Depending on the coupling component used, the products are water-insoluble, metallisable dyes for colouring lacquers, acid or chrome dyes, or substantive, metallisable dyes. Thus, 3-amino-4-hydroxyacetophenone (2 mol.) is diazotised and coupled with the ternary condensation product from 1 mol. of cyanuric chloride, 2 mol. of 2-amino-5-naphthol-7-sulphonic acid, and 1 mol. of aniline, in presence of calcium hydroxide. The disazo dye so formed dyes cotton red by the single-bath coppering method. E. S.

Azo Dyes for Cellulose Acetate, etc. containing *N*-Fluoroalkyl Groups. Eastman Kodak Co. and J. B. Dickey. *U.S.P.* 2,516,302.

Azo dyes for acetate rayon, nylon, etc. are made by coupling a diazo compound with an amine of formula—



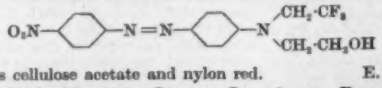
(X = 2:2-difluoroethyl, 2:2-difluoro-*n*-propyl, 3:3-difluoropropyl, 3:3-difluoro-*n*-butyl, or 4:4-difluorobutyl; Y = H or hydroxyalkyl; Z = H, Cl, or CH_3). Thus chlorobenzene is heated in an autoclave with aqueous 2:2-difluoroethylamine in presence of Cu and Cu_2O, to give *N*-2:2-difluoroethylaniline, which reacts with ethylene oxide to give the *N*-2-hydroxyethyl derivative—



Coupled with diazotised *p*-nitroaniline, this gives a red dye for cellulose acetate and nylon.

U.S.P. 2,516,303.

Similar products are obtained by using *N*-trifluoroalkylamines as coupling components (X = 2:2:2-trifluoroethyl, 3:3:3-trifluoro-*n*-propyl, or 4:4:4-trifluoro-*n*-butyl). Thus—



dyes cellulose acetate and nylon red. E. S.

Insoluble Monoazo Copper Complexes—Dyes for Nylon. du Pont, D. E. Kvalnes, and C. Walling. *U.S.P.* 2,517,890.

Dyes capable of dyeing nylon in dark hues from aqueous dispersion are made by coupling diazotised 5-nitro-2-aminophenol with a coupling component having an acyclic group containing a methylene group, in presence of copper compounds, so that coupling and metallisation occur concurrently. Thus, all parts being by weight, the diazo

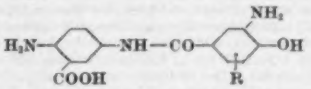
compound from 5-nitro-2-aminophenol (15·4) is poured into a solution of ethyl acetoacetate (13) in water (200) containing ammonia (7) and copper sulphate (30) at 5°c. The product dyes nylon black from a dispersion in soap solution. E. S.

Physical Modification of an Azoic Pigment. American Cyanamid Co. and M. G. Powell. *U.S.P.* 2,517,924.

The light fastness of the azoic pigment *o*-dianisidine→2 mol. of the *o*-phenetidide of 2:3-hydroxynaphthoic acid is much increased by coupling under acid conditions and heating the product at > 100°c. (e.g. at 200°c: in a glass- or nickel-lined autoclave). The desired crystalline form is characterised by an X-ray diffraction spectrum having a line of maximum intensity at an interplanar spacing of 13·3 A. and a line of next greatest intensity at 3·34 A. E. S.

Copperable, Substantive Disazo and Polyazo Dyes. J. R. Geigy A.-G. *B.P.* 649,674.

Yellow and brown copperable substantive dyes are made by coupling the tetrazo compound of a diamine—



(R = H, Hal, CH_3, or O·Alk) either with 2 mol. of β-ketocarboxylic acid derivatives, or with 1 mol. of a derivative of a β-ketocarboxylic acid and 1 mol. of a naphtholsulphonic acid which couples adjacent to the OH group, and is preferably a monosulphonic acid. A preferred coupling component is 1-*p*-aminophenyl-3-methyl-5-pyrazolone.

The diamines of the above type are made as described in *B.P.* 341,970 (J.S.D.C., **47**, 203 (1931)); e.g. by condensing a 3-nitro-4-acyloxybenzoyl halide with 2:5-diaminobenzoic acid, then reducing the NO_2 group to NH_2, and finally saponifying the acyloxy group.

Thus 5-(3′-amino-4′-hydroxybenzoylamino)-anthranilic acid is tetrazotised and coupled with an alkaline solution of 2 mol. of 1-*p*-aminophenyl-3-methyl-5-pyrazolone. The resulting disazo dye gives yellowish browns on cellulosic fibres by the aftercoppering method. E. S.

Brown Trisazo Dyes for Leather. I.C.I. Ltd. and M. Mendoza. *B.P.* 649,313.

Brown leather dyes are made by coupling 2 mol. of diazotised 4-nitro-4′-aminodiphenylamine-2-sulphonic acid with a monoazo compound 1-*m*(or *p*)-aminobenzoylamino-8-naphtholmono(or di)sulphonic acid→a *m*-aminophenol or a *m*-diamine of the benzene series which is capable of coupling twice. Thus 1-*p*-aminobenzoylamino-8-naphthol-3:6-disulphonic acid is diazotised and coupled with *m*-phenylenediamine. The monoazo compound so formed is dissolved in aq. NaOH and coupled with 2 mol. of diazotised 4-nitro-4′-aminodiphenylamine-2-sulphonic acid. Coupling is believed to take place at the 7-position in the naphthalene nucleus and also in the *m*-phenylenediamine residue. E. S.

α-Acylamino-α-hydroxyanthraquinone Mixtures for dyeing Cellulose Esters and Ethers. Ciba Ltd. *B.P.* 648,332.

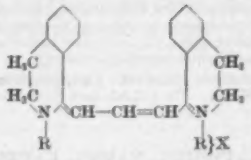
Mixtures of two or more α-acylamino-α-hydroxyanthraquinones, e.g. 1-acetylamino-8-hydroxyanthraquinone together with 1-butyrylamino-8-hydroxyanthraquinone, are ground with an aqueous solution of a dispersing agent or precipitated by pouring a H_2SO_4 solution into water to give compositions which dye cellulose acetate yellows of greater depth than those obtained from either component alone. R. K. F.

Copper Phthalocyanines. Sherwin-Williams Co. and G. M. O'Neal. *B.P.* 648,688.

An *o*-dinitrile other than phthalonitrile itself, e.g. 4-nitrophthalonitrile, is refluxed in nitrobenzene saturated with ammonia in presence of a reaction product of ammonia and cupric acetate to give a copper phthalocyanine pigment. R. K. F.

Cyanine Dyes containing a 3:4-Dihydro*iso*quinoline Nucleus. Eastman Kodak Co., L. G. S. Brooker, and R. H. Sprague. *U.S.P.* 2,525,520.

Dyes of formula—



(R = C_nH_{2n+1} (*n* = 1–4); X = an anion) are obtained by condensing a trialkyl orthoformate with suitable quaternary *N*-alkyl-1-methyl-3:4-dihydro*iso*quinolinium salts. C. O. C.

Toners of Improved Light Fastness. A. F. Schmutzler and D. F. Othmer. *U.S.P.* 2,525,834.

The light fastness of the toners produced by precipitating an acidic solution of a basic dye containing an acidic and a basic group, e.g. Rhodamine B, with a polybasic acid of a heavy metal, e.g. phosphotungstic acid, is improved by heating the precipitated toner. Loss of colour value and change in hue are prevented by adding to the dye liquor or metal salt solution before the precipitation, as an oxidising agent, an inorganic salt containing a heavy metal which will form a complex with the acid group in the dye, e.g. a chromate or dichromate. C. O. C.

Pigment-in-oil Pastes. A. F. Schmutzler and D. F. Othmer. *U.S.P.* 2,525,835.

Oil or other water-immiscible vehicle is added, e.g. as an atomised spray or in emulsion, to the pigment slurry immediately after the pigment has been formed. Both liquids are kept at pH < 7. The product behaves as an oil-free pigment in that it can be filtered and the cake washed free from water-soluble impurities. C. O. C.

V—PAINTS; ENAMELS; INKS

PATENTS

Diminishing Loss of Leafing Power of Aluminium Paste Pigment. Metals Disintegrating Co. Inc. and E. R. Allen. *U.S.P.* 2,525,279.

Alcohols of formula $C_nH_{2n+1}OH$ (*n* = 12–20) are not leafing agents but their addition to aluminium paste pigments diminishes loss of leafing power during storage. *U.S.P.* 2,525,286.

Aliphatic amides of formula $C_nH_{2n+1}\cdot CO\cdot NH_2$ (*n* = 13–20) have the same effect. C. O. C.

Reducing the Tendency of Aluminium Paste Pigments to generate Gas and lose Leafing Power on Storage. Metals Disintegrating Co. Inc. and A. F. Knoll. *U.S.P.* 2,525,301.

Addition of the anhydride or keten of an acid of formula $C_nH_{2n+1}\cdot CH_2\cdot COOH$ (*n* = 12–18) is used. Although these compounds may also be used as the leafing agent, when using them as such it is best not to employ excess over that required for that purpose but to add an additional amount after leafing, 0·1–1·0% depending on the amount of water present in the paste, to act as the protective agent. C. O. C.

Printing Inks leaving Non-tacky Surfaces. J. M. Huber Corpn. and A. Voet. *U.S.P.* 2,525,433.

Inks formed by mixing a pigment in a solution of lignin dissolved in an organic water-miscible solvent, when printed and the solvent removed, either by heating or by contact with water, leave non-thermoplastic films of a particularly dry nature and having little tendency to adhere to similar films under pressure. C. O. C.

Paint for Heavy Coatings. O. V. McGrew. *B.P.* 648,843.

Paint compositions suitable for applying heavy rough-finished coatings ($\frac{1}{8}$ in.) by spraying in one application comprise 18–25% drying oil, e.g. a bodied fish oil, 2–8% fibrous binder, e.g. asbestos, 10–22% of a plate-like material, e.g. ground mica, 10–25% thinner, and the residue of inert filler and/or pigment. E. C.

Solutions of Coal Extracts. C.U.R.A. Patents Ltd. and I. G. C. Dryden. *B.P.* 649,279.

The solutions obtained by treating under atmospheric pressure a bituminous coal with a liquid aliphatic or araliphatic amine containing at least one primary amino group, whose viscosity at the temperature of treatment is

not > 30 centipoises, and then filtering, or aqueous dispersions of such solutions, are used as coating compositions. Applied to metal, glass, etc. and then baked at 250–300°C. they yield hard, water-repellent coatings. C. O. C.

Liquid Cellulose Nitrate Compositions yielding Clear Films. I.C.I. Ltd. and S. F. Marrian. (XI, p. 205.)

VI— FIBRES; YARNS; FABRICS

Use of Lithium Aluminium Hydride in the Study of Carbohydrates. M. Abdel-Akher and F. Smith. *Nature*, **166**, 1037–1038 (16th Dec. 1950).

Lithium aluminium hydride reduces the carbomethoxy group of uronic esters to a primary alcoholic group, the reaction proceeding rapidly at room temp. in inert solvents, e.g. ether or tetrahydrofuran. Methyl esters of, *inter alia*, 2:3:4-trimethyl methyl-D-galacturonoside, 2:3:4-trimethyl α-methyl-D-glucuronoside, and 4-methyl methyl-D-glucuronoside are reduced to 2:3:4-trimethyl methyl-D-galactoside, 2:3:4-trimethyl α-methyl-D-glucoside, and 4-methyl methyl-D-glucoside respectively. The reaction is of wide application in carbohydrate studies, for free sugars and their derivatives can be readily reduced to the corresponding alcohols. J. W. B.

Change with Temperature of the Heat of Wetting of Dry Cellulose in Water, and its Bearing on the Specific Heat of the Adsorbed Water and of the Swollen Cellulose. M. Wahba and S. Nashed. *Nature*, **166**, 998 (9th Dec. 1950).

Heat of wetting of dry standard cellulose, measured between 0° and 40°C., decreases with rise of temperature, the decrease being linear and about 0·042 g.cal./g./°c. This means that the heat capacity of the wetted system is greater than that of the components (cellulose + water it will adsorb) before wetting, which may be due to some change in either water or cellulose. It is suggested that adsorption may cause some depolymerisation of water and, as swelling represents a partial separation of micelles, of cellulose also. J. W. B.

X-Ray Diagram and Ultra-violet Absorption Spectrum of "Renatured" Silk Fibroin. O. Kratky, E. Schauenstein, and A. Sekora. *Nature*, **166**, 1031–1032 (16th Dec. 1950).

An aqueous solution of renatured fibroin, prepared by the method of Coleman and Howitt, is dialysed and dried slowly. Films are obtained, designated silk I, having an X-ray diagram different from that obtained by quick drying (silk II). Moistening with 50% ethanol and stretching up to 300% gives plastic deformation with no orientation, and transformation to silk II does not take place until the moistened films are stretched and rolled at the same time. J. W. B.

Cuticle of Mammalian Hair. H. M. Appleyard and C. M. Greville. *Nature*, **166**, 1031 (16th Dec. 1950).

Transverse and longitudinal sections of human hair, hog bristle, alpaca, and wool are stained and examined under the phase-contrast and normal microscopes, using the 3650 A. line. Cuticles of the first three are comparatively thick, this being due to exposure of only one-sixth of each scale, the remaining five-sixths being covered by adjacent overlapping scale. In a wool fibre, however, the scales, though of similar length, are exposed for five-sixths and overlapped for only one-sixth; hence the cuticle is much thinner. J. W. B.

PATENTS

Spinning Unripe Viscose. American Viscose Corpn. *B.P.* 648,865.

Regenerated cellulosic fibres of high tenacity and extensibility are obtained by extruding viscose, containing 7–8% cellulose, 4–13% sodium hydroxide (x), and a salt point of 8–18 but > 1·9x, into a bath at 45–50°c. containing 1·2–2x% of sulphuric acid and (21 + 0·66x)% of soluble metallic sulphate (> 18% sodium sulphate and > 1% zinc sulphate), and then stretching the fibres 90–110%. W. G. C.

Preventing the Formation of Deposits on Thread-guides, etc. after Viscose Spinning. Courtaulds Ltd., J. Wharton, and R. B. Hall. *B.P.* 649,045.

The formation of hard deposits on thread-guides, godets, or thread-advancing reels during the manufacture of artificial threads from viscose is prevented by treating the thread after leaving the aqueous coagulating bath with an oil, e.g. a mineral lubricating oil, containing an oil-soluble cation-active compound.

Courtaulds Ltd. and J. Wharton. *B.P.* 649,044.

The lubricating oil and the cation-active compound may also be added to the spinning bath. W. G. C.

Casein Fibres for Felting. U.S. Secretary of Agriculture, R. F. Peterson, and R. W. Jackson. *U.S.P.* 2,525,825.

Casein fibres suitable for mixing with fur fibres are produced by washing a casein fibre, formed at pH < 6 and containing a soluble metal salt, in a bath at pH 6–8, hardening and stabilising under tension, lowering the moisture content to 2–4%, and cutting into staple lengths. C. O. C.

Extrusion Spinning of Vinylidene Chloride Polymers. Firestone Tire & Rubber Co. and G. A. Spencer. *B.P.* 649,539.

A continuous rod of vinylidene chloride polymer or copolymer is held between grooved rollers and while in amorphous supercooled condition, between normal temp. and 175°F., is forced toward and through a die and opening of reduced diameter, while simultaneously the rod is drawn through by a further set of rollers to obtain a filament of uniform diameter. The rod may be made slightly plastic by heating providing it can still be compressed. J. W. B.

Cellulose Titanate Films and Fibres. du Pont and F. K. Signaigo. (XI, p. 205.)

Solvent for Polyacrylonitrile. American Viscose Corpn. and M. R. Dalton. (XIII, p. 206.)

Solvents for Aromatic Polyesters. I.C.I. Ltd. and R. May. (XIII, p. 207.)

Solvents for Acrylonitrile Polymers. American Viscose Corpn. (XIII, p. 207.)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Studies in the Bleaching of Jute. I— Action of Sodium Hypochlorite. W. G. Macmillan, A. B. S. Gupta, and S. K. Majumdar. *J. Indian Chem. Soc., Ind. & News Ed.*, **12**, 105–112 (1949): *Chem. Abs.*, **44**, 11,105 (25th Nov. 1950).

Jute cloth treated for 90 min. in aq. NaOCl (5 g. chlorine per litre) is readily bleached if the solution is alkaline, the colour improving with increase in Na_2CO_3 content up to 10 g./litre, beyond which there is no improvement. Bleaching is inferior in acid or neutral solution, even though the Cl_2 consumption is higher. In acid solutions Cl_2 consumption is high, bleaching is poor, loss in weight is high, too much lignin is removed, substances other than lignin are removed, and tensile strength is poor. High loss of lignin in acid hypochlorite may be due to formation of soluble chlorolignin compounds, whereas with alkaline hypochlorite oxidation with a much diminished degree of chlorination may occur. Pretreating the cloth with NaOH reduces the Cl_2 consumption, probably because of the removal of soluble products which if left in the cloth would consume much Cl_2. With both untreated and NaOH-treated cloth Cl_2 consumption and lignin removal increase with increase in concn. of Cl_2 and with a progressive improvement in the bleach. Cloth not pretreated with NaOH is difficult to wet out in the NaOCl soln. Alkali pretreatment results in a more uniform bleach, which is, however, tinged yellow. A better bleach is given if Calsolene Oil HS (ICI) is added to the bleach liquor instead of giving an NaOH pretreatment. The solution recommended is available Cl 7, Na_2CO_3 10, and Calsolene Oil 2·5 g./litre. A reasonably good jute bleach is obtained by repeated 90-min. treatments (about six) with a more dil. NaOCl soln. containing 3 g. available Cl per litre. Cl_2 consumption, high in the initial treatments, decreases with the number of treatments. C. O. C.

Bleaching Reactions with Jute Fibre. H. J. Callow. *Nature*, **166**, 1040 (16th Dec. 1950).

Partial acetylation of jute with a mixture of acetic anhydride and pyridine, as well as methylation with an ethereal solution of diazomethane, retards the rate of bleaching. For example, a lignin-free fraction may be isolated from raw jute after three chlorinations and

sulphite extractions, whereas an acetylated sample requires up to seven such treatments. Colour reactions associated with lignin after acid chlorination and addition of sodium sulphite are much less marked with acetylated material, but are restored by cold dilute alkaline hydrolysis. Comparison is also made of the bleaching rates, and colour reactions, of acetylated jute before and after exposure to mercury-vapour irradiation. No change in either is observed, thus showing that the acetyl groups which are removed by irradiation are not those primarily responsible for retarding the bleaching reactions of acetylated jute. J. W. B.

PATENT

Single-process Dry Cleaning. United States Hoffman Machinery Corpn. *B.P.* 649,785.

Goods are cleaned in an air-tight rotary-drum tumbler by rotating at relatively low speed, drained, and centrifuged at a speed insufficient for harmful vibration; residual solvent is removed *in vacuo* by bringing in live steam at 0·5–2 lb./sq. in., thus permitting removal of solvent below 110°F. Rotation is continued at a speed adequate to ensure thorough contact between steam and garments, the temp. being sufficiently low to prevent fixing of stains and the final moisture content being suitable for subsequent pressing. J. W. B.

VIII—DYEING

PATENTS

Dyeing Plastic Articles or Fibres with an Aqueous Dispersion of Dye dissolved in a Plasticiser. Interchemical Corpn. and L. F. Koberlein. *U.S.P.* 2,524,811.

Infinite dilution with water of dye solutions in water-immiscible solvents, without either separation of the dye or breaking of the solvent dispersion, is obtained by use as dispersing agent of a long-chain alkali-treated sulphonated oil which has been treated with a lower aliphatic amine or with morpholine. C. O. C.

Dyeing of Carpets *in situ*. R. G. Dixon & Co. Ltd. and A. F. Bruce. *B.P.* 649,476.

The dye liquor is applied to the pile of the carpet *in situ* and is worked in by brushing. This may be done by means of a shampooing machine in which the tank for detergent solution has been replaced by one having an electric immersion heater so that the dye liquor can be boiled. C. O. C.

IX—PRINTING

Degradation of Viscose Rayon in Reduction Discharge Printing. J. Pinte, Pierret, and Rochas. *Bull. Inst. Textile France*, No. 15, 43–62 (1949): *Chem. Abs.*, 44, 11,107 (25th Nov. 1950).

Tendering and degradation of viscose rayon by Na formaldehyde-sulphoxylate in discharge printing are due to presence of air during steaming. This reducing agent acts as a catalyst for oxidation of cellulose by atmospheric oxygen. This catalytic action disappears after the first 5 min. of steaming; damage does not increase after this time. The degradation caused is of the periodate type, and its extent clearly defined by cupric ethylenediamine fluidity. Damage to cotton under similar conditions is attributed to the same cause. C. O. C.

PATENTS

Light-sensitive Diazonium Salt Materials. L. L. Ridgway Co. Inc., P. E. Spoerri, and D. P. Habib. *U.S.P.* 2,525,751.

A diazo salt of 2:3-dihydroxynaphthalene-6-sulphonic acid is used as the light-sensitive layer for photographic paper. C. O. C.

Diazotype Photoprinting Materials sensitised with Diazosulphones. General Aniline & Film Corpn., J. A. Sprung, and W. A. Schmidt. *B.P.* 649,604.

Compounds of formula $R^1{\cdot}N{:}N{\cdot}SO_2{\cdot}R^2$ (R^1 = subst. or unsubst. aryl or heterocyclic radical; R^2 = subst. or unsubst. aryl or heterocyclic radical, subst or unsubst. Alk or cycloalkyl radical) are used. When unexposed to light, they readily couple with azo dye components in presence of an alkaline medium. C. O. C.

X—SIZING AND FINISHING

Behaviour of Cellulose in Alkalis. J. Cyrot and R. Petit. *Bull. Inst. Textile France*, No. 14, 9–20 (1949): *Chem. Abs.*, 44, 11,087 (25th Nov. 1950).

Sorption of NaOH by cellulose is unaffected by temp. $> 40°$C. if correction is made for water evaporation from the lye. Below 40°c. there is decrease in sorbed NaOH. Likewise, increase of time of treatment beyond 30 min. has no effect. There is no evidence for the existence of a definite alkali cellulose compound. C. O. C.

PATENTS

Relaxing Chlorinated Wool Fabric. A. Guttman and D. Guttman. *U.S.P.* 2,525,543.

Excellent dimensional stability is imparted to chlorinated wool fabric by tumbling it in water at 75°F. and drying, preferably by tumbling in air at 120°F. C. O. C.

Reducing the Degradative Action of Light on Nylon. I.C.I. Ltd., R. G. Desson, H. R. Hadfield, and L. Wood. *B.P.* 649,481.

Not $> 1\%$ by weight of a compound containing cationic chromium is incorporated into nylon, e.g. by treating first in an acidified aqueous solution of a dichromate and then with a reducing agent so as to form chromic ions. C. O. C.

Rendering Cellulosic Materials Dimensionally Stable by Treatment with Divinyl Sulphone. United States Rubber Co., D. L. Schoene, and V. S. Chambers. *U.S.P.* 2,524,399.

Cotton and regenerated cellulose rayon fabrics are rendered stable to shrinkage on laundering by treatment with vinyl sulphone in presence of a basic catalyst. C. O. C.

Crushed Velvet resistant to Wetting. T. Van Heek. *U.S.P.* 2,524,119.

Crushed velvet is rendered insensitive to repeated thorough wettings by heating it in either water or a moist atmosphere at as much above 140°F. as the material will stand whilst it is still in the tightly wound or twisted rope form in which it is after crushing. C. O. C.

Stabilising Fabrics containing Rayon. Stein, Hall & Co. Inc. and F. G. La Piana. *U.S.P.* 2,524,113.

Fabrics containing rayon can be stabilised by the action of 10–50% aq. alkali (at 50–100°F. for 30–600 sec.), and if desired rendered creaseproof and given any desired crinkling and two-tone dyeing properties, by treating them with a protective material before treating with the alkali. Suitable protective agents are resins which become insolubilised by the action of alkali; e.g. the fabric is treated with a 3–25% aq. soln. of the condensate of an aldehyde, an α-hydroxymonocarboxylic acid, an α-substituted ethanol, and either urea, thiourea, guanidine, carbamylurea (biuret), or an aminotriazine containing at least one primary amine group, and dried at 200°F. before passing to the alkali bath. C. O. C.

Flameproofing of Cellulosic and Protein Materials. Joseph Bancroft & Sons Co. *B.P.* 648,883.

An aqueous solution of an acid of phosphorus and/or sulphuric acid and/or sulphamic acid, a non-metallic compound containing N and C, an aldehyde of < 5 C, and a fugitive neutralising agent is used. The non-metallic compound should be basic with respect to the acid and the C : N ratio should be less than that of C : N in biuret, e.g. urea. The neutralising agent must be removable by heat, and sufficient should be used to bring the solution to pH 6–8, the proportions of the other ingredients being such as to produce (in absence of the neutralising agent) a pH corresponding to a mixture of orthophosphoric acid with 1–10 mol. of urea and 0·2–1·5 mol. of formaldehyde. After impregnation with this solution, the material is dried and baked at 250–400°F. for 30 sec.–2 hr., and then washed free of soluble products. This gives a non-toxic, durable, odourless finish without affecting the original handle of the material, and so is particularly applicable to fabrics intended for garments. C. O. C.

Imparting Flame- or Mildew-resistance to Textiles. Joseph Bancroft & Sons Co. *B.P.* 649,642.

Cellulose and/or protein textiles are given durable flame-resistance by impregnating them with an aqueous solution, or the reaction products, of (1) a non-volatile phosphorus or sulphur acid containing no metal, organic group, or

constituents yielding active O or Hal, or a metal-free salt of such an acid which decomposes during the process to yield the acid and a volatile cation, and (2) a non-metallic nitrogenous organic compound soluble in and basic towards the acid, its atomic C : N ratio being not $> 2 : 1$. The amount of acid and nitrogen applied to the fibre should be such that the final complex obtained contains 1–5% P or S and 0·25–6% N on its dry weight. The impregnated material is dried and then baked at 400–250°F. for 30 sec. to 120 min., when it should have pH 3–6. Finally it is washed and dried. C. O. C.

Artificial Leather from Vinyl Polymers. Ciba Ltd. *B.P.* 649,972.

A material greatly resembling leather in internal structure, elasticity, resistance to tearing, handle, and appearance is prepared by heating and pressing, e.g. through a calender, an uncompressed fleece of loose fibres such as carded wool, carded cotton or both, or several such fleeces, with a foil (or several such fleeces interspersed with foils) of polymerisate, e.g. highly polymeric polyvinyl compounds, preferably containing softening agents such as dibutyl phthalate or tricresyl phosphate. By pressing the top layer of the bonded fibrous mass with a cooled embossing roller a leather-like grained surface is produced. J. W. B.

Restoring the Nap on Worn Cloth which has become Smooth or Glazed. E. F. Struckman. *B.P.* 649,979.

Grit is blown through the cloth under controlled air pressure so that it cuts or severs the entwined fibres while the air blast combs the separated fibres. C. O. C.

Permanently Sized Fabrics. General Aniline & Film Corpn. and C. Spatt. *B.P.* 647,273.

A finish resistant to washing is imparted to fabrics, particularly those of vegetable origin, by impregnating them with a mixture of a polyhydric alcohol and a synthetic resin obtained by copolymerisation of maleic anhydride with a polymerisable compound containing one $>C{:}CH_2$ group. The mixture should have pH 3·0–4·5. Finally the impregnated fabric is heat-cured. C. O. C.

Coated Fabrics capable of Transmitting Water Vapour. Wingfoot Corpn. *B.P.* 649,094.

A waterproof water-vapour-permeable fabric is obtained by coating cloth with a continuous layer of a natural or synthetic rubber composition deposited from a liquid dispersion and from 2–6 vol. (based on the rubber) of a hydrophilic diatomaceous earth. C. O. C.

Coating by means of Thermoplastic Transfer Sheets. Sylvania Industrial Corpn. *B.P.* 648,812.

A transfer sheet is prepared by coating a temporary backing sheet, e.g. an endless aluminium band, with a resin-forming compound or precondensate which is normally liquid and has a lower softening point than the backing sheet, for which it has no chemical and/or physical affinity. It is then treated so that the liquid polymerises to form a solid film capable of being transferred to another surface by heat and pressure. Such a transfer sheet is especially suitable for producing light-weight flexible waterproof materials, e.g. for coating nylon fabric with phenol–furfuraldehyde resin. C. O. C.

Waterproof Coatings. Société des Usines Chimiques Rhône-Poulenc. *B.P.* 647,713.

Waterproof coatings exhibiting a large angle of contact with water are obtained by application of a fine inert powder, e.g. silica or kaolin, which has been waterproofed by treatment with an organosilicic derivative. The powder is bonded to the surface to be coated by means of an adhesive, preferably by being embedded in a heat-hardened organosilicic resin, the respective amounts being such that the coating has a fine-grained surface. C. O. C.

Coating Webs. J. Waldron Corpn., C. A. Dickhaut, and B. R. Newcomb. *B.P.* 647,886.

Excess of a coating composition is applied at one point in the path of the web and is then forced into closer contact with the web so as to increase the adhesion, excess being removed by impinging a jet of air at such an angle that the component of force tending to drive the composition into the web exceeds the component tending to blow the surplus backward. The surplus is thus atomised, and is intercepted to prevent its subsequent redeposition on the web from the ambient air. The composition is applied to the underside of the horizontal moving web, which passes round a platen roller as it meets the air jet. The underside may be pre-wetted and doctored before the composition is applied. The apparatus comprises a frame carrying a platen roller, a nozzle tube supported on an axis parallel to the roller and having a longitudinal slot for directing a jet of air against the roller, a baffle having a ridge adjacent to the periphery of the roller and parallel to its axis, and means for projecting the air jet at an angle between 90° and 45° to the tangent plane to the platen roller at the line of contact. S. V. S.

Laminated Sheet Material. American Viscose Corpn. *B.P.* 648,816.

A non-woven layer of fibres some or all of which are thermoplastic is bonded by heat and pressure to a film of thermoplastic organic material under such conditions that the film material does not impregnate the layer of fibres. The products are especially suitable as light-weight materials for tents, raincoats, tarpaulins, etc. C. O. C.

Elastic Fabrics. United States Rubber Co. *B.P.* 648,644.

A highly shrinkable composite yarn is formed by twisting together a non-thermoplastic yarn and a much smaller, heat-shrinkable continuous resinous filament. An open or loosely constructed fabric is made partly or wholly from this yarn, and is then heat-shrunk. A stretchable rubberlike sheet is then bonded to the shrunk fabric, and finally the coated fabric is treated to remove the resinous filament. The product is capable of being stretched to an extent fixed by the non-thermoplastic yarn remaining in the fabric. C. O. C.

Impregnating Keratinous Filaments. John R. Evans & Co. and W. Windus. (XII, p. 206.)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Stain for Use in the Microscopy of Beaten Fibres. F. L. Simons. *Tappi*, 33, 312–314 (1950): *Chem. Abs.*, 44, 11,089 (25th Nov. 1950).

When fibres are stained with a mixture of the blue *C.I.* 518 and the orange *C.I.* 621, bruised spots, broken ends, fibrils, and fibre débris are coloured orange whereas unbeaten fibres are coloured blue. 1% solutions of each dye are used. These are mixed in equal proportions for wood pulps, and 45% blue : 55% orange for rag pulps. Fibres are dried on a slide; 6–8 drops of stain are added, and the fibres reduced at 60–70°c. The fibres are washed on the slide with water, dried, and examined. No mounting is necessary. C. O. C.

PATENTS

Coated Paper Sheets. A. Szwarc. *B.P.* 649,261.

A kraft paper sheet has at least one side coated with a composition containing, as essential body-forming constituents in total amount of at least about 99% by weight of the composition, 50–80% of asphalt mineral rubber (elaterite) of m.p. 320–350°F., 10–25% of a mixture of polymerised coumarone and polymerised indene (the coumarone and indene being obtained from solvent naphtha fractions of coal-tar at 280–340°F.), 2–10% of a methyl ester or hydrogenated methyl ester of rosin, and 4–7% of paraffin wax of m.p. 122–150°F. The composition may include 0·5–1% copper naphthenate as a fungicide. S. V. S.

Impregnation of Paper Felt with Hot Tar. Koppers Co. Inc. and M. G. Bigley. *B.P.* 649,767.

Paper felt is passed through a bath of hot coke-oven tar containing naphthalene and then into an enclosure kept above the sublimation temperature of naphthalene. Thence it is withdrawn into an atmosphere at $< 60°F.$ The temperature is maintained in the enclosure by continuously exhausting the atmosphere from it with simultaneous admission of air at not $< 60°F.$ C. O. C.

De-inking Waste Paper. du Pont and F. L. Fennell. *U.S.P.* 2,525,594.

Printed waste paper is readily de-inked by treating it with an aqueous alkaline solution containing alkali-metal peroxide and then rinsing. There is decreased shrinkage and the de-inked pulp requires less severe bleaching to produce a good grade of paper. C. O. C.

Processing Fibrous Pulp for Papermaking. Price Brothers & Co. Ltd. *B.P.* 647,905.

The pulp is rolled transversely, in the presence of an aqueous liquid, in different directions between two

opposed working surfaces while it is under compression by and in tractive contact with these surfaces, at least one of which is perforated, allowing liquid to flow out from the stock. Liquid may also be introduced into the pulp during treatment, which is continued until a desired fraction of the finer constituents or soluble matter has been removed with the liquid leaving through the perforated surface. Ink particles e.g. may be liberated from the fibres and carried away in water, which may contain a detergent and a bleaching agent. The compression of the stock may be increased until the consistency reaches a certain value, the liquid leaving through various ducts at different rates. If both working surfaces are perforated, different liquids may be introduced in different parts of the traverse, and withdrawn at other parts. A regular cyclical variation in mutual clearance is imparted between the working surfaces as they are moved relative to one another in different tangential directions relative to nodules or aggregates of nodules of pulp. The machine for this treatment has opposed parallel perforated working surfaces, basically plane but having numerous small pyramidal protrusions on them and separated by a narrow clearance. A gyratory movement is imparted to one surface, so that all points in the moving surface travel relative to the other surface in circles of equal diameter. S. V. S.

Heat - sealable, Moistureproof, Transparent Regenerated Cellulose Sheeting. British Cellophane Ltd. *B.P.* 647,593.

A non-fibrous base of regenerated cellulose is coated with a mixture of a ketone–formaldehyde resin and a melamine–formaldehyde resin, which is insolubilised after application and is then coated with a moistureproof, heat-sealable cellulose nitrate coating. C. O. C.

Cellulose Ethers and Esters and Synthetic Resins plasticised with a Bis(chlorobutoxybutyl) Phthalate. Celanese Corpn. of America and G. J. Shugar. *U.S.P.* 2,525,961.

Compounds of formula—

$$Cl\cdot[CH_2]_4\cdot O\cdot[CH_2]_4\cdot O\cdot CO\cdot C_6H_4\cdot CO\cdot O\cdot[CH_2]_4\cdot O\cdot[CH_2]_4\cdot Cl$$

are excellent solvents and plasticisers for cellulose acetate and other cellulose derivatives and for certain synthetic resins. C. O. C.

Cellulose Titanate Films and Fibres. du Pont and F. K. Signaigo. *U.S.P.* 2,525,049.

Regenerated cellulose film or fibres when treated with a solution of a titanium salt so that the titanium forms a chemical compound with the cellulose, e.g. by treatment with aq. $TiOSO_4$, have their opacity to ultra-violet radiation much increased and their degradation on exposure to sunlight and sensitivity to moisture much reduced. C. O. C.

Liquid Cellulose Nitrate Compositions yielding Clear Films. I.C.I. Ltd. and S. F. Marrian. *B.P.* 649,694.

Liquid triesters of aliphatic tricarboxylic acids in which all three ester groups contain mono-ethylenically unsaturated aliphatic alcohol radicals are solvents for cellulose nitrate. On subjecting a solution of cellulose nitrate in one or more of these esters to polymerising, and if desired oxidising, conditions, the cellulose nitrate remains in solution in the resinous polymerised, or polymerised and oxidised, insoluble forms of the triesters. C. O. C.

Coating Webs. J. Waldron Corpn., C. A. Dickhaut, and B. R. Newcomb. (X, p. 204.)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Charge of the Leather Surface. I—Rôle of the Charge in Tanning and Aftertanning, II—Rôle of the Charge in Dyeing and Fat Liquoring. G. Otto. *Leder*, 1, 81–86; 105–107 and 133–137 (1950): *Chem. Abs.*, 44, 11,143 (25th Nov. 1950).

I—The ability of leather to react with polar substances is determined by its surface charge. The isoelectric point of limed collagen is 4·9–5·2; above this pH it reacts with anions and below it with cations. Vegetable tannins are weak acids that combine with the hide by secondary valency. Vegetable-tanned leather has a negative surface charge and therefore attracts the cations of basic dyes, but can be dyed with anionic dyes if enough acid is used to bring the pH below the isoelectric point. Mineral-tanned leather usually has a positive charge, although the leather may take up either complex anions or cations from the tanning bath depending on the salts used in its preparation. Mineral-tanned leather must be treated with an anionic substance, e.g. a vegetable or synthetic tanning agent or an acid dye, before dyeing with a basic dye. Neutralising chrome leather reduces its positive charge, so that anionic dyes or fat emulsions are absorbed more slowly and consequently more evenly. In retanning chrome leather with a vegetable tanner the latter rushes on unevenly unless a mild tannin, e.g. gambier or sumac, is used or the positive charge of the leather is reduced by treatment with sulphonated oil or acids that form stable complexes with the chromium salt.

II—If the surface charge of the leather is too great, the dye tends to rush on unevenly. This can be corrected by adding to the dyebath a product having colourless ions with charges of the same sign as the dye ions. Syntan-dyed leather has a strong negative surface charge but can be dyed with anionic dyes if the positive groups of the collagen are first charged by lowering the pH. To obtain even take-up of dye the acid should be added after the first addition of dye. Tamol (Basf), a neutral salt of tanning sulphonic acids having colourless ions, is designed as an assistant in applying anionic dyes to chrome leather. There are two types—(1) an acid dye type for pastel dyeings, in which sulpho groups predominate and fixation is through principal valencies, and (2) a substantive dye type which is fixed by secondary valencies. Chromatography has shown that dyewood extracts contain both coloured and colourless ions. Fat liquors are usually negatively charged emulsions and so are unstable in acid solutions; products with soluble cations are now made, usually containing quinquevalent N. Use of the proper agents enables the fat at choice either to be deposited on the leather surface or to penetrate evenly to the centre. C. O. C.

Chemistry of Chromium in Chrome Leather. G. Otto. *Leder*, 1, 153–156 (1950): *Chem. Abs.*, 44, 11,149 (25th Nov. 1950).

Leather tanned with basic chromic sulphate and dried is difficult to wet with water. Neutralising chrome leather with complex-forming buffer salts results in formation of anionic complexes, and the same change occurs during drying, as is shown by the fact that after either process there are—(1) uniform dyeing with pure anionic dyes, (2) development of affinity for basic dyes, and (3) reduction in the reactivity of the chromium with chrome dyes. C. O. C.

The Theory of Chrome Tanning. S. G. Shuttleworth. *J. Soc. Leather Trades Chem.*, 34, 410–437 (Nov.) and ix (Dec. 1950).

The adsorption, residual valency, and salt formation theories of chrome tannage do not provide satisfactory explanations of the thermal stability and resistance to rapid leaching by mineral acids of chrome-tanned leather. Theories of bridge formation by co-ordination of both amino and carboxyl groups of the leather are shown to be based on a misinterpretation of spectrophotometric evidence. Conductimetric methods indicate that neither amino nor peptide groups can co-ordinate with chromium under chrome tannage conditions, and spectrophotometric data confirm this. Kinetic experiments are described which show that tanning consists primarily in co-ordination of the protein carboxyl groups to the chrome complex. High thermal stability is explained by the formation of bridges between collagen carboxyl groups by an olated chromium atom. The rules governing the behaviour of masking agents in chrome tannage are deduced from conductimetric data. A. S. F.

Soaking of Australian Dried Woolskin. M. Dempsey. *J. Soc. Leather Trades Chem.*, 34, 464–473 (Dec. 1950).

Pieces of dried merino sheepskin soaked for 1, 2, 4, and 8 days in water, water containing zinc chloride as disinfectant, 10% aqueous calcium chloride, and sulphuric acid at pH 2 were examined for efficiency of soaking by studying the resultant fibre structure. Water-soaked samples soon showed bacterial damage, and while this was inhibited by zinc chloride, a considerable time was necessary in order to open up the structure. Soaking with calcium chloride was accompanied by loss of substance, whereas the use of acid led to a plump, compactly woven structure. A. S. F.

Aging of Karakul and Seal Fur Skins. E. T. Steiner and E. R. Hosterman. *Bur. Stand. J. Res.*, **45**, 317–322 (Oct. 1950); *Research Paper* 2141.

Physical and chemical data show that deterioration takes place in Karakul and seal fur skins when stored under conditions not expected to be injurious. Pelts weakened by aging show increased soluble nitrogen and water-soluble contents, but lower petroleum ether-soluble contents than less aged skins. The presence and quantity of copper in the weakened pelts offer the most likely explanation of the cause and extent of deterioration. This is in agreement with previously published work on the influence of copper and iron on vegetable-tanned leather caused to undergo accelerated deterioration. A. S. F.

PATENTS

Dehairing of Hides by Electrolysis. United Shoe Machinery Corpn. and R. A. Whitmore.
U.S.P. 2,523,486.

An electrolytic solution of pH 7, e.g. 1% NaOH in 30% methanol, is applied to the hair side only of a wet untanned hide, which is then spread out and pressed between two electrodes. An electric current is passed through the hide, preferably from the flesh to the hair side, one of the electrodes being free of the electrolytic solution. The hair may then be easily scraped from the hide without appreciable injury to either hide or hair. C. O. C.

Tanning of Hides. E. G. Wilson. *B.P.* 648,236.

A machine is described in which the leather, immersed n tanning liquor, is bent repeatedly from end to end, thus constantly ejecting and absorbing the liquor from and into the leather. This shortens the time needed for tanning and yields leather of improved quality. C. O. C.

Production of White Leather by Retanning Chrome-tanned Leather with a Sulphonic Acid Syntan and then with a Hydroxymethylmelamine. American Cyanamid Co. and W. O. Dawson.
U.S.P. 2,522,666.

Chrome leather is given greatly improved whiteness and light-resistance while maintaining its physical properties by treating it first with an aromatic sulphonate tanning agent and then with a water-soluble melamine–formaldehyde condensate or hydroxymethylmelamine. C. O. C.

Tanning in Non-aqueous Medium. United Shoe Machinery Corpn. and R. C. Putnam.
U.S.P. 2,523,324.

Leather, which withstands prolonged soaking in hot water without marked shrivelling or hardening and is exceptionally resistant to mould growth, is obtained by bringing the skin to pH 5–7, drying to 10–15% moisture content, and then treating with a 10–50% solution (by vol.) of a chloroformate, e.g. glycol bischloroformate, in an organic solvent.
U.S.P. 2,523,325.

The tanning liquor used consists of a 10–50% solution (by vol.) of an acid chloride of a C_2–C_{12} fatty acid, a dichloride of a C_3–C_{12} dibasic acid, phenoxyacetyl chloride, *m*-sulphobenzoyl dichloride, trichloroacetyl chloride, or chloroacetyl chloride in the formal of the mono-methyl or -ethyl ether of ethylene or diethylene glycol.
U.S.P. 2,525,326.

A 10–65% soln. (by vol.) of a C_5–C_{12} aliphatic *iso*cyanate, chloromethyl *iso*cyanate, hexamethylene di*iso*cyanate, or 2:4-tolylene di*iso*cyanate in an organic solvent is used as the tanning liquor. C. O. C.

Preserving Hides and Pelts. C. H. Gibson.
U.S.P. 2,525,539.

Instead of the usual salting and pickling, the hides are drummed with water at 28–30°c., and then 1·5% salt and 1·4–2·8% slaked lime are added; the drum is again rotated, then 2·8–5·6% Na_2S added, and the drum rotated for 20 min. 1 per mill [*sic*] of a yeast and 5% wheat waste are then added, and the hides drummed for 20 min., and then drummed for 20 min. with running water at 28–32°c. The flow of water is stopped 1 min. before the end of the drumming, 2·5% $NaHSO_3$ added, and drumming resumed for 30 min., after which the hides are washed with water. On bating, the hides are very compact, do not lose weight, are immune to attack by rats, moth or mildew, and do not deteriorate on long keeping however the temperature may change. The hides do not require liming or beam-house operations before they can be classified by the tanner, as in the dry dehaired state of the hides all defects can be seen at a glance. C. O. C.

Impregnating Keratinous Filaments. John R. Evans & Co. and W. Windus. *B.P.* 649,974.

Modification of *B.P.* 628,989 (cf. J.S.D.C., **66**, 163 (Feb. 1950)). Keratinous filaments, e.g. untanned sheepskins or furs, are treated with an acidic solution containing free resorcinol or pyrogallol and a free aldehyde reactive therewith, e.g. formaldehyde, benzaldehyde, furfuraldehyde, or their polymers. When furs, etc. are treated by this method, the skins are tanned and the hair is rendered thermoplastic, so that it can be straightened at a fairly high temperature and on cooling will not readily revert to its previous woolly or kinky condition. The hair is also coloured, usually reddish or brownish but sometimes bluish. It is also rendered more resistant to heat, alkalis, and wear. The lustre of the treated fibres is increased, and in the case of furs and imitation furs where the fibres (hair or wool) are still attached to the skin the products show a closer nap and hence give the appearance of a greater hair or wool count. C. O. C.

Synthetic Tanning Agents from Aromatic Sulphonic Acids. L. Bolgár. *B.P.* 649,516.

Sulphonic acids of novolaks are condensed with urea (and/or thiourea)–ammonia–aldehyde condensates containing active hydroxymethyl and amino groups.
B.P. 649,570.

Using β-naphtholsulphonic acid instead of a sulphonated novolak yields a product which has a bigger filling capacity. C. O. C.

XIII—RUBBER; RESINS; PLASTICS

PATENTS

Imitation Leather. Underfelts Ltd. and A. Kemp. *B.P.* 648,331.

A fine aqueous suspension of precipitated rubber latex is mixed with an aqueous dispersion of disintegrated fibrous material, after which water is removed from the mixture, which is then formed into sheets. C. O. C.

Linoleum, etc. having Variegated or Marble-type Graining throughout its Thickness. Armstrong Cork Co. and S. H. Hartman. *B.P.* 649,043.

A laminated sheet composed of > 2 layers of plastic composition, at least two of the layers being of different colours, is cut into small laminated sections, which are then pressed heterogeneously together to form a continuous sheet by passing them through a sheeting calender. C. O. C.

Velvety Surfaces on Flexible Plastic Bases. G. Bernstein. *B.P.* 649,120.

A layer of natural or synthetic fibres is distributed on an auxiliary moving surface which deposits them on a sheet of plastic composition as it emerges hot from a calender, the whole being compressed by a subsequent cylinder. If desired, the plastic-backed velvet surface may be applied to textile fabrics. J. W. B.

Calendering Chlorosulphonated Polyethylene. du Pont. *B.P.* 648,485.

Chlorosulphonated polyethylene can be calendered to form sheets by incorporating 2–5% of a polyoxyethylene glycol, e.g. Carbowax 1500 (Carbide & Carbon Chemical Corpn.). W. G. C.

Polyvinyl Alkyl Ethers stabilised to Heat. General Aniline & Film Corpn. and C. E. Schildknecht.
U.S.P. 2,521,950.

Addition of 0·2–2·0% by weight of *p*-hydroxy-*N*-phenylmorpholine and enough of an alkali-metal hydroxide, carbonate, or bicarbonate to give pH 9 stabilises polyvinyl alkyl ethers to discoloration and depolymerisation on heating. C. O. C.

Solvent for Polyacrylonitrile. American Viscose Corpn. and M. R. Dalton. *U.S.P.* 2,523,282.

A mixture of 60–80% by volume of nitromethane and 40–20% of *a*-hydroxypropionitrile does not swell homopolymers of acrylonitrile of mol. wt. $> 15{,}000$ but does completely dissolve them at 80–90°c. The solutions are very clear, colourless, and homogeneous. They may be used as lacquers and coating compositions, for preparing films and fibres, etc. C. O. C.

Phenol–Aldehyde and Epoxide Resin Compositions. Devoe & Raynolds Co. Inc. and S. O. Greenlee. *U.S.P.* 2,521,911.

Compositions containing a phenol–aldehyde condensate having reactive hydroxyl groups and a complex, solid epoxide resin, which is a polyether derivative of a polyhydric alcohol containing epoxy groups and free from functional groups other than epoxy and hydroxyl groups, become insoluble and infusible on heating. They have wide uses as coatings, films, etc.

U.S.P. 2,521,912.

The ratio of epoxide groups of the polyepoxide to phenolic hydroxyls in the phenol–aldehyde condensate is between 1·8 : 1 and 1 : 1·4, and there is an alkaline catalyst present. C. O. C.

Aliphatic and Araliphatic Azo Compounds—Polymerisation Catalysts. du Pont and J. A. Robertson. *U.S.P.* 2,520,338.

Aliphatic azo compounds of formula—

$YOOC{\cdot}CHR^1{\cdot}CH_2{\cdot}CR^2(CN){\cdot}N{:}N{\cdot}CR^2(CN){\cdot}CH_2{\cdot}CHR^1{\cdot}COOY$

(Y = a univalent cation; R^1 = H or a hydrocarbon radical of 1–6 C; R^2 = a hydrocarbon radical of 1–6 C) are catalysts in the polymerisation of compounds containing ethylenic double bonds. Thus, by reaction of sodium laevulate with hydrazine hydrate, followed by treatment of the product, first with HCN, then with chlorine, $\alpha\alpha'$-azobis(α-methyl-γ-carboxybutyronitrile) is formed (Y = H, R^1 = H, R^2 = CH_3). This, mixed with 1% vinyl acetate, acts as a catalyst in the polymerisation.

U.S.P. 2,520,339.

Araliphatic azo compounds Ar·CHR·N:N·CHR·Ar (R = hydrocarbon radical of 1–10 C) act similarly as polymerisation catalysts. Thus, acetophenone and hydrazine react together, and the resulting azine is hydrogenated in presence of Pd to give the hydrazo compound, which is dissolved in *n*-heptane and oxidised with oxygen gas to 1:1′-azobis(1-phenylethane) (Ar = C_6H_5, R = CH_3). This may be used in the polymerisation of e.g. ethylene. E. S.

***cyclo*Pentadiene Resins.** Velsicol Corpn. and C. F. Peters. *B.P.* 648,892.

Copolymers of *cyclo*pentadiene and an unsaturated ester, e.g. vinyl acetate or dimethyl maleate, are prepared by heating them at 390–575°F. under pressure. The resinous products are compatible with cellulose nitrate and/or drying oils, and are suitable for use in lacquers and varnishes. E. C.

Vinylidene Chloride and Vinyl Chloride Polymer Compositions of Improved Heat Stability. Dow Chemical Co., E. C. Britton, and W. J. Le Fevre. *U.S.P.* 2,525,643.

Vinylidene chloride or vinyl chloride polymers or their copolymers with one another or with other compounds in which there is at least 10% of one of these chlorides are given improved stability to heat by adding to them 0·5–5% by weight of α-amino-α-phenylacetic acid. C. O. C.

Dispersions of Vinyl Chloride Resins. Carbide & Carbon Chemicals Corpn., R. W. Quarles, and C. I. Spessard. *B.P.* 649,609.

Mixtures of lower glycol ethers with liquid aromatic hydrocarbons are solvents for low-mol.wt. vinyl chloride resins and also have the proper swelling action on vinyl chloride resins of higher mol. wt. to form stable fluid organosols. The ether : hydrocarbon ratio at which the more fluid compositions are obtained is nearly the same in both cases. They may be used to prepare coating, etc. compositions containing both dissolved and suspended vinyl chloride resins of low and high mol. wt. C. O. C.

Plasticiser for Vinyl Resins. B. F. Goodrich Co. *B.P.* 648,900.

Polymers and copolymers of vinyl or vinylidene chloride are plasticised with a copolymer (mol. wt. $\leq$ 85,000) of 1:3-butadiene (30–80%) and acrylonitrile or its α-substituted derivatives (7–20%). E. C.

Solvents for Aromatic Polyesters. I.C.I. Ltd. and R. May. *B.P.* 649,655.

Esters of tetrahydrofurfuraldehyde dissolve aromatic polyesters, e.g. Terylene, to clear, pourable solutions of high concentration. C. O. C.

Solvents for Acrylonitrile Polymers. American Viscose Corpn. *B.P.* 649,474.

α-Chloro-β-hydroxypropionitrile has a good solvent action on polyacrylonitrile of mol. wt. approx. 200,000 at 70°c., with complete dissolution at 100°c. The solutions remain stable indefinitely if kept below 100°c., gelation occurring only very slowly below, and not at all above, 50°c. C. O. C.

Curing Butadiene–Acrylonitrile Copolymers. I.C.I. Ltd., D. H. Coffey, O. B. Edgar, T. J. Meyrick, and J. T. Watts. *B.P.* 649,928.

Organic poly*iso*cyanate-modified polyesters are not only excellent curing agents for butadiene–acrylonitrile copolymers but also act as excellent extenders, which do not readily volatilise or migrate from the compositions. Such compositions are useful *inter alia* in leather cloth manufacture. C. O. C.

Kinetics and Mechanism of Copolymerisation. A. D. Abkin. *Doklady Akad. Nauk S.S.S.R.*, **75**, 403–406 (21st Nov. 1950).

Acetone Resins for Laminating Paper, Cloth, etc. Bakelite Corpn. (III, p. 197.)

Artificial Leather from Vinyl Polymers. Ciba Ltd. (X, p. 204.)

Cellulose Ethers and Esters and Synthetic Resins plasticised with a Bis(chlorobutoxybutyl) Phthalate. Celanese Corpn. of America and G. J. Shugar. (XI, p. 205.)

XIV—ANALYSIS; TESTING; APPARATUS

Determination of the Calcium and Magnesium Hardness in Water by Direct Colorimetric Titration. J. G. Walker and V. Murtagh. *Water*, **55**, 8–10 (Jan. 1951).

The titrimetric method of Schwagenback, Biedermann, and Bangerter (*Helv. Chim. Acta*, **30**, 1303, 1798 (1947); **31**, 331, 456, 678, 1029 (1948)) for the separate determination of the Ca and Mg contents of a water has been tested on a series of natural waters. It was found to be simple, rapid, and consistently accurate to within 2% when checked by gravimetric methods. The permissible limit of concentration of some of the more common ions was determined by trials on artificial waters. Because of the high permissible chloride content, the method should be particularly useful for evaluating base-exchange materials. C. O. C.

Determination of Oxygen in Dirty Washing Liquors. E. Walther. *Fette & Seifen*, **52**, 478–482 (1950): *Chem. Abs.*, **45**, 2241 (10th March 1951).

2 ml. of 0·4% $Ti_2(SO_4)_3$ in conc. H_2SO_4 is added to 10 ml. of the washing liquor and compared with a series of standards prepared from H_2O_2. C. O. C.

Determination of Anionic Synthetic Detergents in Sewage. H. C. Evans. *J.S.C.I.*, **69**, S 76–S 80 (Supplementary Issue No. 2, 1950).

Although small concentrations of synthetic anion-active detergents may be estimated by extracting the active material as the methylene blue complex from the aqueous solution by chloroform and measuring the colour intensity of the extract, active material in urine and some inorganic ions, e.g. nitrates and thiocyanates, which may be present in sewage, are also extracted. However, the extraction of synthetic anion-active detergents is not affected by changes in the pH of the aqueous solution, whereas extraction of the other substances is decreased as the pH of the solution is lowered. This difference is made the basis of a method for the determination of synthetic anion-active detergents in sewage. The amount of active material determined at pH 3·25 and 0·7 is plotted against pH, the true detergent content being given by a continuation of this line to pH −2·0. The method has been used successfully on two widely different types of sewage and added alkyl sulphates have been correctly estimated. W. K. R.

Scheme of Analysis for Commercial Detergents—I. J. A. Gilby and H. W. Hodgson. *Mfg. Chemist*, **21**, 371–376 (1950): *Chem. Abs.*, **45**, 371 (10th Jan. 1951).

A critical review with 46 references of the principal methods of analysis for anionics, cationics, and non-ionics. From established principles a modified Linsenmeyer (*Chem. Abs.*, **35**, 2635 (1941)) analytical scheme is set up, with the active ingredient only, by which the following

classes of material are identified separately—soaps; sulphonated oils; protein fatty acid condensation products; ethylene oxide condensation products; alkyl sulphates, alkyl sulphate monoglycerides, fatty acid esters of sulphoacetamides, alkylaryl polyether sulphonates; and alkylarylsulphonates. To confirm and establish the fine structure of the active agents, the Lassaigne test was used to divide the products according to their ultimate analysis into four groups—(1) Only C, H, and O present indicates soaps and non-ionics. (2) Only S present indicates alkylsulphonates, alkyl sulphates of monoglycerides, alkylarylsulphonates, and alkylaryl polyether sulphonates. (3) Both N and S present indicates fatty acid esters of sulphoacetamides. (4) N and/or Hal present indicates mainly cationics. C. O. C.

Identification of Tylose in Detergents and Identification of Surface-active Substances. E. Weber. *Fette & Seifen*, **52**, 477, 478 (1950): *Chem. Abs.*, **45**, 2241 (10th March 1951).

To test for surface-active agents in presence of Tylose or other polysaccharide, 1–2 g. of the detergent is acidified with conc. H_2SO_4 and covered with 3–5 ml. conc. H_2SO_4, and the mixture extracted with chloroform. In presence of Tylose the extract is violet and changes to yellow when made alkaline. This test is not obtained when the surface-active agent present is Nekal BX or an alkylphenylsulphonate; in such case some soap or alkylsulphonate is added before the test. In absence of Tylose the chloroform extract is colourless. To identify the surface-active agent, 2 ml. conc. H_2SO_4 is poured down the side of a test-tube containing 1 ml. of a 1% aq. soln. of the surface-active agent and 1 ml. of a 1% aq. soln. of the Tylose; a ring of characteristic colour and fluorescence (under ultra-violet radiation) forms at the interface. The test can also be carried out by mixing 1 ml. each of the 1% solutions and 3% conc. H_2SO_4, standing for 30 min., observing the colour in daylight and in ultra-violet radiation, extracting with formaldehyde, and noting the colour of the extract. The following colours are given (compound, daylight, ultra-violet radiation, chloroform extract); soap—ruby red turning brick red, grey–violet, none or pale red; alkylsulphonates—brick red, grey–violet, none; fatty alcohol sulphates—red–violet, blue–violet, violet; Nekal BX—dirty green, olive green, none; alkylphenylsulphonates—yellowish brown, dirty yellow–green, none; mineral oil—no reaction; C_7–C_9 satd. acids—brown, brown, none; $C_{17}H_{35}$·COOH—brown, brown, none; satd. alcohols of > 6 C—red–violet, violet, violet; unsatd. acids—wine red, wine red, wine red. C. O. C.

Rapid Determination of Aniline. A. I. Bulycheva. *Zavodskaya Lab.*, **14**, 1208, 1209 (1948): *Chem. Abs.*, **44**, 10,603 (25th Nov. 1950).

A method based on colorimetry of the reaction of aniline with chloramine T in alkaline solution in presence of phenol (recommended 3% phenol in 2% NaOH). A blue builds up in 15–20 min. and is stable for 2–3 days. This blue is compared visually with a calibrated set of standards. Determination of aniline in the atmosphere is readily done by separation into a water trap; 92–100% recovery and determination are possible with samples containing as little as 0·01–0·2 mg./litre. C. O. C.

Colorimetric Determination and Chromatographic Separation of *s*-Trinitrobenzene and *m*-Dinitrobenzene. K. Cruse and R. Mittag. *Z. anal. Chem.*, **131**, 273–278 (1950): *Chem. Abs.*, **45**, 500 (25th Jan. 1951).

s-Trinitrobenzene can be determined accurately by means of the red complex it forms with sodium ethoxide in benzene solution. *m*-Dinitrobenzene can likewise be determined in ultra-violet radiation. When both are present, they can be separated chromatographically using freshly activated MgO in benzene. C. O. C.

Report on [Determination of] Colouring Matters in Foods. C. F. Jablonski. *J. Assocn. Off. Agric. Chem.*, **33**, 517–521 (1950): *Chem. Abs.*, **45**, 775 (25th Jan. 1951).

A method for determining FD&C Yellow No. 5 in presence of FD&C Yellow No. 6 has given satisfactory results in collaborative study. It is based on the complete destruction of No. 6 by H_2S when heated under pressure, No. 5 being only slightly affected. C. O. C.

Vanadium Number. M. V. Gapchenko. *Zavodskaya Lab.*, **16**, 1126, 1127 (1950): *Chem. Abs.*, **45**, 2209 (10th March 1951).

With some dyes there are difficulties in titrating with $TiCl_3$. Replacement of the latter by V permits titration at room temperature in absence of Rochelle salt and the colour change is very clear. NH_4VO_3 is dissolved in *N*-H_2SO_4 and then reduced by Zn–Hg. C. O. C.

Analysis of Phenolphthalein. S. F. Popov. *Med. Prom. S.S.S.R.*, (6), 25, 26 (1949): *Chem. Abs.*, **45**, 500 (25th Jan. 1951).

Dissolve a 0·1 g. sample in 15 ml. 0·1 *N*-NaOH, add 8–10 ml. 0·5 *N*-$KBrO_3$ and 5 ml. 10% KBr, and, while shaking, rapidly add 25 ml. acid mixture (20 ml. 80% acetic acid and 5 ml. conc. HCl); after shaking for 1 min., add 1 g. KCl and titrate the liberated iodine with 0·1 *N*-$Na_2S_2O_3$. One ml. 0·1 *N*-$KBrO_3$ = 0·003977 g. phenolphthalein. Run a blank in parallel. C. O. C.

Permanganate as a Spraying Reagent for Amino-acids on Paper Chromatograms. C. E. Dalgliesh. *Nature*, **166**, 1076 (23rd Dec. 1950).

If a paper chromatogram carrying amino acids is lightly sprayed with potassium permanganate reagent (a 1% solution containing 2% sodium carbonate), methionine, tryptophan, tyrosine, and (more weakly) proline and histidine immediately appear as yellow spots on a red background. The spots should be marked at once, because as the paper dries the colour difference tends to disappear. Threonine, hydroxyproline, lysine, and glycine show up on standing a few minutes. The reagent is not applicable when phenol has been used as irrigating solvent. J. W. B.

Some Further Observations on the Analysis of Nylon Type Polymers. J. Haslam and M. Clasper. *Analyst*, **76**, 33–40 (Jan. 1951).

Methods are presented for the analysis of mixtures of hexamethylenediamine dihydrochloride (I) and 5-aminocaproic acid hydrochloride (II), which may be obtained on hydrolysing certain nylon-type interpolymers with 50% HCl (*cf.* J.S.D.C., **66**, 100 (Jan. 1950)). II is determined by titration with standard alkali, using phenolphthalein as indicator. On passing a solution of the mixed hydrochlorides through a column of Amberlite IRA 400 resin, the efflux consists of I, which is determined by titration with standard alkali, using methyl orange as indicator. The results of an examination of the hydrolysis products o polymers produced (*a*) by interaction of hexamethylene di*iso*cyanate and 1:4-butanediol and (*b*) from ω-amino-undecanoic acid are described. W. K. R.

Quantitative Analysis of Wool–Silk Mixtures with Cupric Ethylenediamine. J. Pinte and Mlle. Jafflin. *Bull. Inst. Textile France*, No. **12**, 23–28 (1949): *Chem. Abs.*, **44**, 11,106 (25th Nov. 1950).

Wash successively in two cupric ethylenediamine solutions and then in HCl (25 g./litre), finally rinsing to neutrality. The residue of wool is obtained on a sintered glass filter, dried, and weighed. This weight should be multiplied by 1·02 to get the actual weight of wool in the sample. The first cupric ethylenediamine soln. contains 0·70% Cu and 1·39% ethylenediamine, the second soln. contains 0·35% Cu and 0·69% ethylenediamine. C. O. C.

Separation of Silk–Nylon Mixtures and Determination of the Two Constituents. Mlle. Jafflin. *Bull. Inst. Textile France*, No. **13**, 19–21 (1949): *Chem. Abs.*, **44**, 11,106 (25th Nov. 1950).

Wash first in an aq. soln. containing 0·70% Cu and 1·39% ethylenediamine, then in one containing 0·35% Cu and 0·69% ethylenediamine, and finally in HCl (10 g./litre). This dissolves out the silk and leaves the nylon. C. O. C.

Measuring the Whitening Effect of a Detergent. A. Lassieur. *Bull. mens. ITERG* (*Inst. tech. Études et Recherches Corps gras*), **4**, 520–526 (1950): *Chem. Abs.*, **45**, 2239 (10th March 1951).

Critical review of usual colorimetric and gravimetric methods for measuring the efficiency of a detergent. An economical photometric apparatus consists essentially of a light source and condenser as used in photographic enlarging, a photoelectric cell, and an 18-v. galvanometer. The cloth to be examined is subjected to a thin beam of light, and part of the light diffused is directed to the photoelectric cell. The intensity of the current set up is proportional to the degree of whiteness. C. O. C.

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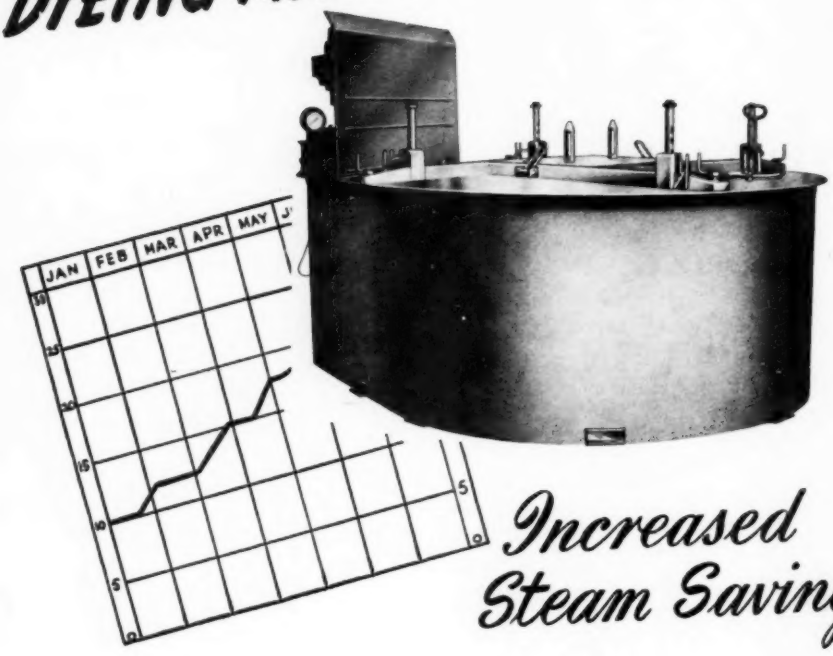

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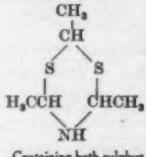

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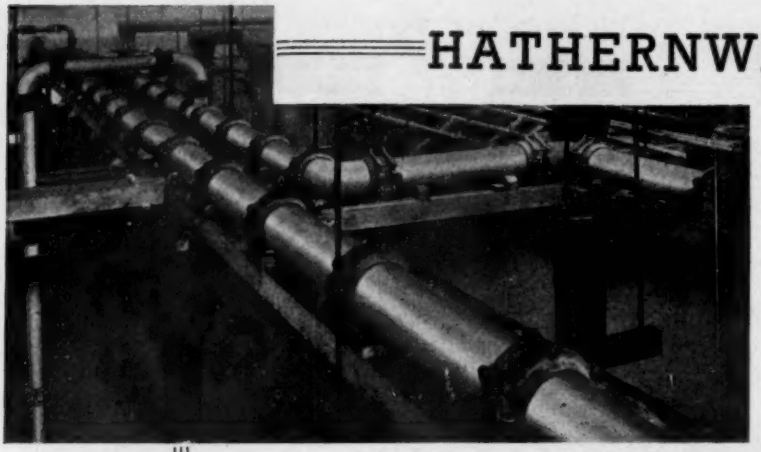

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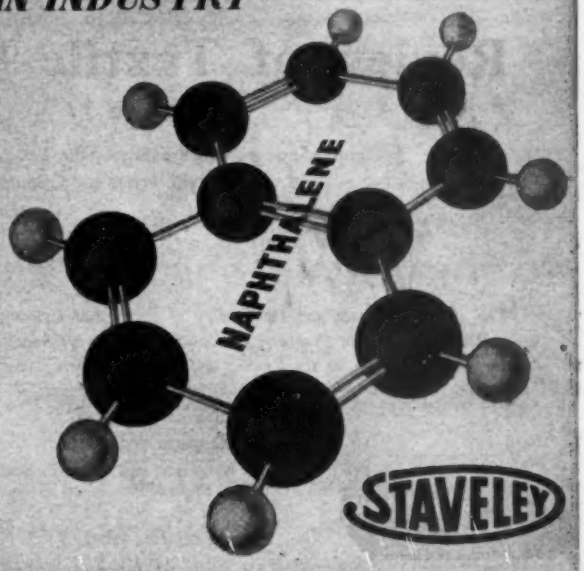

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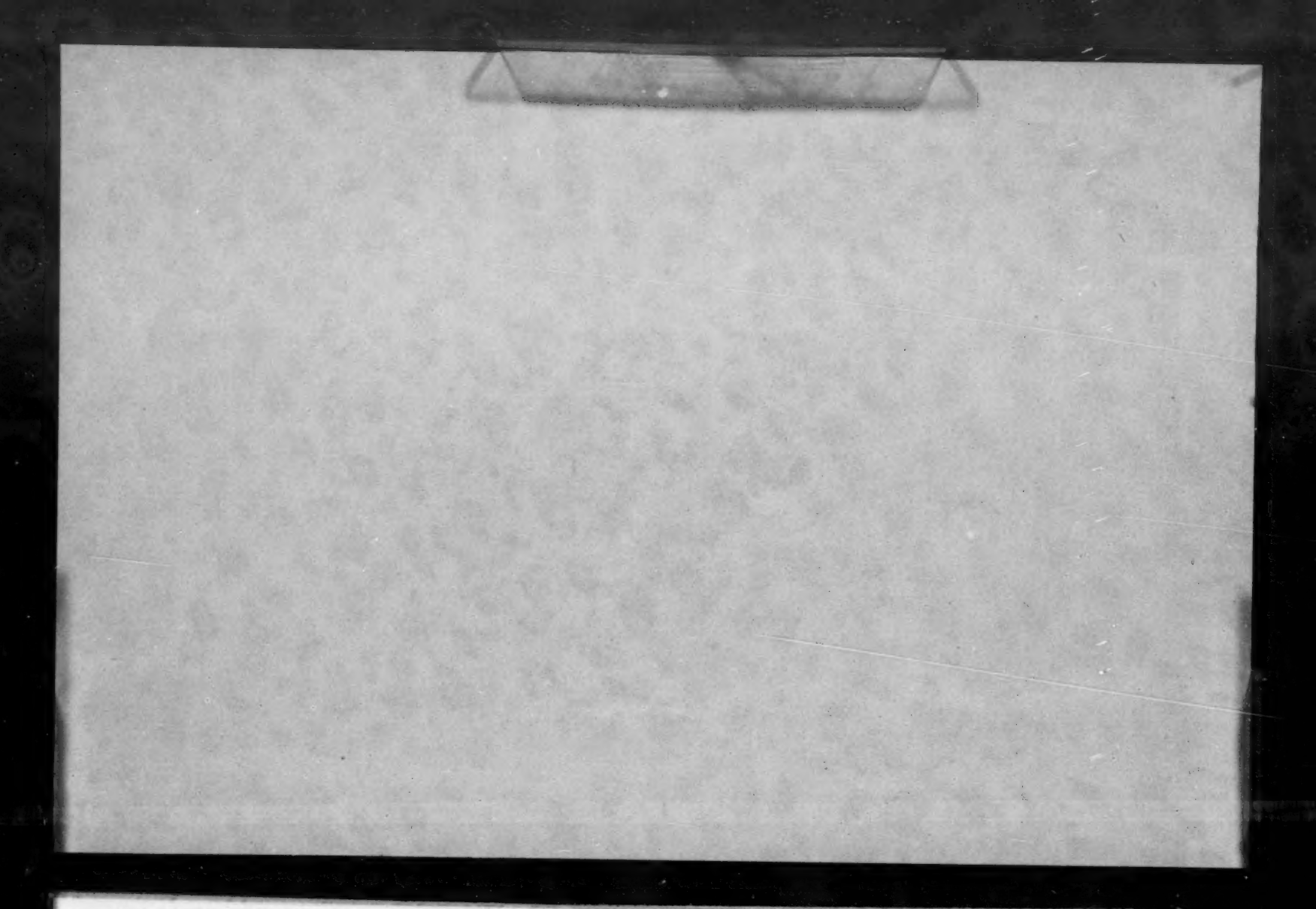

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